PETROLEUM AND NATURAL GAS RESEARCH PROGRAM BUREAU OF MINES, FISCAL YEAR 1950

BY R. A. CATTELL AND OTHERS

* * * * * * * * * * **Information Circular 7616**

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by

R. A. Catlell[1] and others

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SUMMARY AND INTRODUCTION

The Bureau of Mines recently observed its fortieth anniversary, and with the close of the fiscal year 1950 it completed 36 years of close cooperation with and helpfulness to the petroleum and natural-gas industries, whose technical problems it studies. Petroleum people likewise observed the ninetieth anniversary of the start of this truly American industry when "Colonel" Drake drilled the first well at Titusville, Pa., in 1859 (see fig. 1). Throughout the period, during which there have been many changes in the fuel and related economy of the country, this work has been carried on systematically by an organized entity of the Bureau first known as the Petroleum Division and in recent years as the Petroleum and Natural Gas Branch. Changes in the industries have been brought about partly by economic forces, partly by advances in technology, and in some measure by the impact of two world wars. As the needs of industry and the Nation have changed, so have the studies of the Bureau changed to meet these needs.

Consistently, Bureau technologists and scientists have tried to keep in step with progressive movements; frequently, in fact, have taken the lead. Wherever possible, they have looked far ahead in their program of research, at the same time recognizing the needs of the hour.

In the preceding annual report,2/ a brief backward glance was taken at the 5 year period, 1945-1949. This action was helpful in that it served to draw an intermediate base line from which to view critically more recent accomplishments. The 1949 report emphasized that great advantage accrues to industry and Government by joining hands "in a wise use of technical and scientific knowledge gained through research." The same pattern has been maintained in the work for the fiscal year 1950 reported in the following pages.

The emphasis of that work has been on fundamentals, and in general the research has been basic. As the year was characterized by relatively stable conditions, it was possible to plan and conduct research on the major projects of a long-range program without the disruptions that attended the war years, when the emphasis was on obtaining quick results on special assignments.

Administratively, the Bureau of Mines was regionalized. However, this shift from a centralized organization (reporting directly to Washington) to one of decentralization (with petroleum and natural-gas activities conducted in four of the Regions of the Bureau of Mines) has not changed the national continuity of the main technical program. The work now, as formerly, is industry-wide. For example, the

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2/ Cattell, R. A., and others, Report of the Petroleum and Natural Gas Branch, Fiscal Year 1949: Bureau of Mines Inf. Circ. 7551, 1950, 50 pp.

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results of secondary-recovery research at Franklin, Pa., are as applicable in parts of Texas as in Pennsylvania and the areas immediately adjacent. In fact, during the year many oil-well cores were collected in Oklahoma and Texas fields and delivered to the Franklin laboratories for study. Later they will be given further tests in the applied physics laboratories of the Eastern Experiment Station, College Park, Md. All of this initial field work and resulting laboratory research is building up new factual data regarding the use of explosives for shooting oil and gas wells throughout the country.

The study of sulfur compounds in petroleum under A.P.I. Research Project 48A is another excellent example of the integration of work conducted at widely separated headquarters. The synthesis, purification, and determination of properties of sulfur compounds at Laramie, Wyo., necessarily must go hand in hand with the separation, isolation, and identification and with the determination of thermodynamic data of sulfur compounds at Bartlesville, Okla. This fundamental approach to a problem as old as the discovery of high-sulfur crude oil in the Lima, Ohio, field in 1885 is pointing out truths regarding the composition and characteristics of these deleterious substances that were never known before and that will aid refiners throughout the world.

The foregoing examples illustrate the general situation pertaining to a maintained, coordinated national program with each problem chosen and assigned to a major project because its solution adds one more essential member to the framework of a well-planned structure of fundamental knowledge. The time has long passed when an engineer or chemist can choose at will any problem that seems attractive without first canvassing the situation carefully to ascertain whether or not it is an integral part of the research program.

The paragraphs that immediately follow briefly point to a few of the more important accomplishments under this national program. It will be quickly evident that the experimental work and basic thinking leading to the results or pointing the way to additional exploratory endeavor could only have been done in an environment that is conducive to orderly and progressive development of technical knowledge.

The research on reservoir-oil samples offers a good example of a closely knit project that is an integral part of the primary production-research program of the Bureau. Using data obtained from previously reported work on samples from such reservoirs as those in the North Lindsay and Elk City fields, Okla., and similar data from the literature on reservoirs containing large quantities of gas in solution, engineers of the Petroleum Experiment Station, Bartlesville, Okla., have shown that estimates of ultimate recovery of oil should include the amount of liquid hydrocarbons that will be condensed from "free" or liberated gas that enters the well bore. Generally used methods, as reported in the literature, do not consider this factor. The result has been that estimates of the ultimate recovery from reservoirs containing highly volatile oils may be in error as much as 100 percent. The full import of this work is not yet known, but the knowledge elicited may have a direct influence on the development and producing procedures applied to many reservoirs containing highly volatile oils. The net effect would be a revision upward of the estimated petroleum reserves of the country and a stimulation of interest in the development of reservoirs of this type because of the larger anticipated return.

During the fiscal year 1950, increased emphasis was placed on the fundamental research project on the nature of surface forces in petroleum reservoirs, conducted as an integral part of the secondary-recovery program. At the beginning it was made

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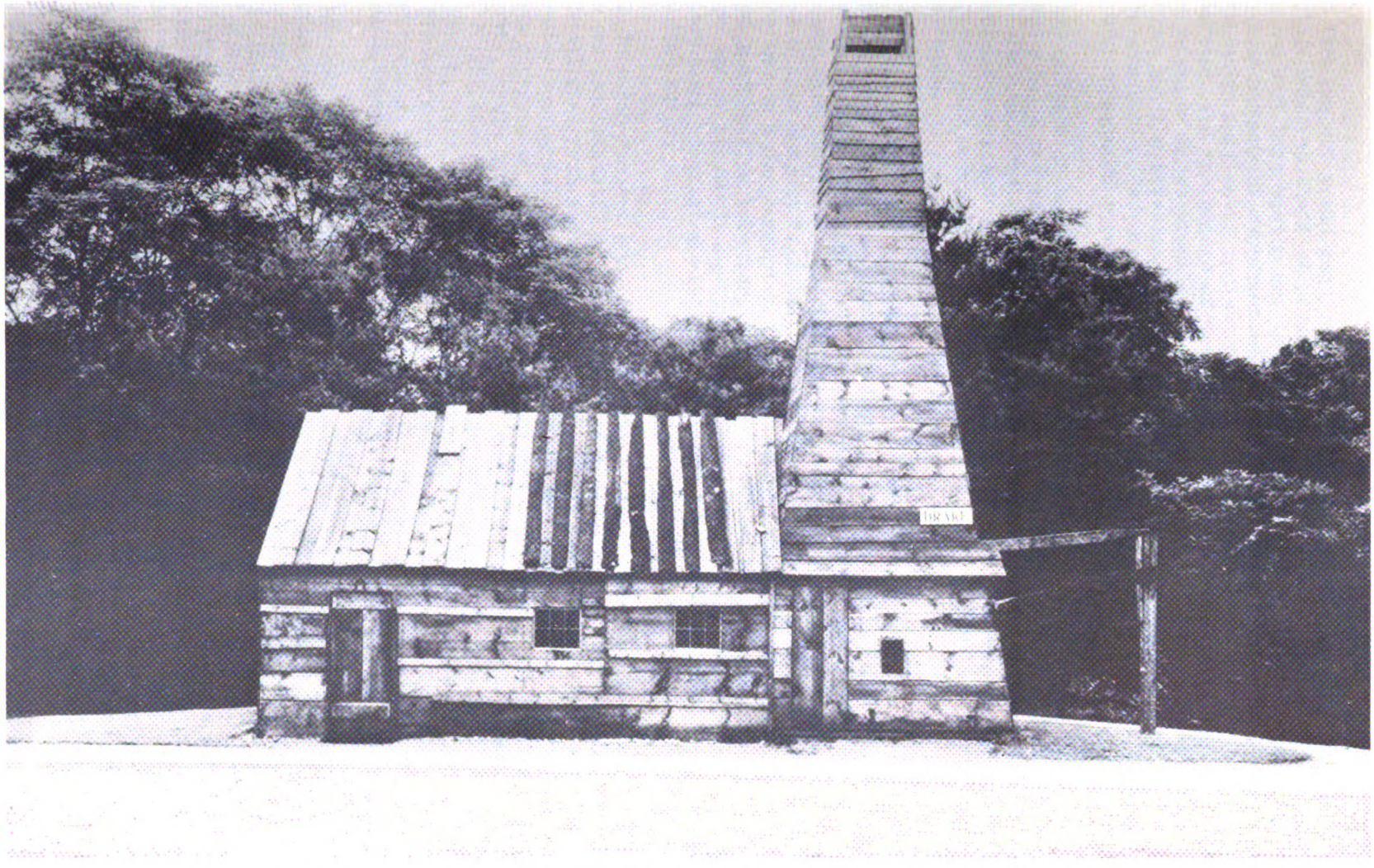

Figure 1. - Replica of world's first oil well - the Drake well, Titusville, Pa., 1859. (Courtesy Paul H. Giddens, Curator, Drake Well Memorial Park, Titusville, Pa.).

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clear that this project would require uninterrupted, painstaking, and patient research by a carefully selected staff of physicists and chemists. It was pointed out, also, that these men must have vision and imagination that will take them beyond conventional, routine methods of experimentation and oil-production practices.3/ In 1950 the program was in full swing, including studies of the fluid-fluid interfaces, liquid-solid interfaces, and the nature of the "surface-active" constituents of crude petroleum. The Bureau of Mines stoutly maintains that it is not in search of startling "discoveries", but the research has gone far enough to prove that the data being obtained are contributing factors in solving the industry-old problem of recovering more oil from underground reservoirs.

Another evidence of basic research along unconventional lines was the recent groundbreaking for a new radiochemical laboratory at Bartlesville. This compact structure, when completed, will be used in the study of radioisotopes as tracers in water-flooding and air and gas repressuring. The Bureau has well-qualified personnel to conduct this work under the general approval of the Isotope Division of the Atomic Energy Commission.

The long-range, balanced program of the Bureau dealing with chemistry and refining not only emphasizes the composition of petroleum and its products but also the combustion of hydrocarbon fuels. Representatives of industry and of the military establishments have displayed and continue to display keen interest in the fundamental studies of this subject that the Bureau scientists and engineers are making. Much of the work is being conducted in a constant-volume bomb to obtain fundamental information on combustion behavior, using test samples of 5 cc. or less of both pure hydrocarbons and of conventional-type fuels. The results of this small-scale experimentation are being correlated with data obtained from test engines used in rating commercial-type fuels.

A.P.I. Research Project 48A, dealing with sulfur compounds, is exemplary of the wide scope and applicability of the results of the Bureau's program. Under this project, distillates from approximately 20 high-sulfur crude oils were analyzed for types of sulfur compounds, resulting in the most extensive survey concerning the distribution of types of sulfur compounds that has been made. Based on the results of this survey, crude oil from Wasson, Tex., was selected for more intensive investigation. Distillates from this crude oil, prepared by isothermal distillation at low temperatures, were treated with solid adsorbents, such as alumina, and the sulfur compounds present were concentrated into only 2 to 3 percent of the original distillate. Concurrently, experiments were conducted to determine relative adsorptivities of the various types of sulfur compounds on solid adsorbents.

To study the temperature stability of naturally occurring sulfur compounds, sulfur-bearing crude oils and their distillates have been subjected to prolonged heating periods, and the evolved hydrogen sulfide and mercaptans measured. These measurements indicate whether the more complex sulfur compounds are decomposing and the extent of the decomposition. As a corollary, experiments on the thermal decomposition of pure sulfur compounds are going forward. Successful separation and identification of sulfur compounds in crude oils depend largely on having available suitable analytical techniques, which, in turn, are based largely upon the chemical behavior and physical properties of pure sulfur compounds. Accordingly, pure sulfur compounds are being prepared and their optical and physical properties determined. Infrared and ultraviolet spectra are particularly useful in the identification of the compounds separated from crude oil.

3/ Fowler, H. C., Secondary Recovery Research and the Bureau of Mines: Interstate Oil Compact Quarterly Bull., vol. VII, No. 1, May 1948, pp. 19-24.

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An important part of the petroleum thermodynamics work of the Bureau of Mines pertains directly to this project. Conditions necessary for the decomposition of sulfur compounds by heat and their transformation to other types of sulfur compounds will be predictable when thermodynamic data are available. Pure sulfur compounds are being studied to provide the complete range of thermodynamic properties required. Despite the difficulties resulting from the corrosive nature of the substances, techniques have been developed for measuring accurately the vapor heat capacity, heat of vaporization, vapor pressure, and entropy of these substances. The compounds studied are referred to more specifically in the body of this report, where thermodynamic research on pure hydrocarbons also is described. Many of the experimental data obtained have been correlated and their usefulness extended to high temperatures by the methods of statistical mechanics. The development and construction of a rotating bomb calorimeter of radically new design for determining accurately the heat of combustion of sulfur compounds is an important contribution to calorimetric techniques.

Despite the seeming tranquillity that contributes to the helpful environment in which to conduct research of the types described in this report, there has been no cessation of alertness to the needs of national security. Those in charge of the work have continually reviewed their programs and the work on problems in progress critically to see whether they meet munition and fuel needs or how they may best be adapted to those needs.

The individual problems now being studied are discussed under the following captions: Oil and gas development and production, Secondary recovery, Petroleum chemistry and refining, Thermodynamics of petroleum, Transportation of natural gas, and Helium tracer studies. These projects combine to make up the petroleum and natural-gas program of the Bureau of Mines and closely related phases of the helium-research program. The work done by the Bureau on health and safety in the petroleum industry, on petroleum economics and statistics, in other phases of research on helium and helium-bearing natural gas, and in conservation and production of helium are reviewed in other writings and will not be discussed in this report.

ACKNOWLEDGMENTS

This account of the petroleum and natural-gas program and accomplishments is based on reports prepared by the scientific staffs of six field headquarters in four of the Bureau's nine regions. Under the new organization, personnel of the Petroleum and Natural Gas Branch in Washington serve on the Director's staff, and personnel in the field are under the supervision of regional directors. As a result, titles of all head supervisors of the field headquarters dealing with oil and gas research and with closely related helium research were changed. This acknowledgment of their valuable assistance in preparing the reports takes account of those changes and lists the regional directors of the four regions in which the work was performed:

H. C. Miller, Regional Director, Region III,
San Francisco, Calif.

G. B. Shea, Chief, Fuels Technology Division, Region III,
Petroleum Field Office, San Francisco, Calif.

J. H. East, Jr., Regional Director, Region IV,
Denver, Colo.

H. P. Rue, Chief, Fuels Technology Division, Region IV,
Petroleum and Oil Shale Experiment Station,
Laramie, Wyo.

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C. W. Seibel, Regional Director, Region VI,
Amarillo, Tex.

H. C. Fowler, Chief, Fuels Technology Division, Region VI,
Petroleum Experiment Station, Bartlesville, Okla.

K. F. Anderson, Acting Chief, Engineering Field Studies Branch,
Region VI, Petroleum Field Office, Dallas, Tex.

P. V. Mullins, Chief, Helium Division, Region VI,
Bureau of Mines Helium Plants, Amarillo, Tex.

H. P. Greenwald, Regional Director, Region VIII,
Pittsburgh, Pa.

S. S. Taylor, Chief, Petroleum and Natural Gas Branch,
Region VIII, Petroleum Field Office, Franklin, Pa.

The Bureau of Mines has received splendid cooperation from the petroleum and natural gas industry, and grateful acknowledgment is made to the industry for the assistance given to many of the research projects described in this report. In the fiscal year 1950 the work was given further substantial assistance by means of 16 cooperative agreements, which resulted directly in increased accomplishments. For this the Bureau of Mines makes grateful acknowledgment and expresses appreciation to the following cooperators: State of Oklahoma, Kansas State Board of Health, Arkansas Oil and Gas Commission, University of Wyoming, Texas Technological College, American Petroleum Institute, Natural Gas Department of American Gas Association, Coordinating Research Council, Inc., Pennsylvania Grade Crude Oil Association, and North Texas Oil and Gas Association.

The photograph showing the replica of the Drake Well, figure 1 in this report, was furnished through the courtesy of Paul H. Giddens, curator, Drake Well Memorial Park, Titusville, Pa.

OIL AND GAS DEVELOPMENT AND PRODUCTION

Characteristics of Gas-Condensate Fluids

A monograph entitled "Phase Relations of Gas-Condensate Fluids" is in preparation that will stress the correlations of phase-relationship data obtained by use of the mobile laboratory at flowing wells in the field. As interpretation of these data is technically complex, more than one correlation is being prepared, so that users of the data may choose between a simple and easily applied correlation providing approximate accuracy and more complex correlations providing maximum accuracy. The simplest and most readily applied correlation requires information concerning the liquid:gas ratio at the wellhead pressure and at the temperature of the flowing fluid of unknown characteristics. This correlation provides pressure, temperature, and liquid:gas ratio information for the fluid in commonly used engineering units. By means of the correlation, a complete phase diagram can be computed for the fluid covering the range of pressures and temperatures significant in production practice. The most accurate correlation is one involving pressures and temperatures expressed in units of critical-state pressure and temperature and liquid:gas ratios expressed as a fraction of the liquid-product content of the flowing fluid.

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A search of the literature provided a summary of properties of hydrocarbons occurring in gas-condensate fluid that is based on the most reliable fundamental data now available. Vapor pressures, critical constants, vapor-liquid phase equilibrium factors, and partial liquid volumes have been selected and tabulated for normal paraffin hydrocarbons, containing up to 25 carbon atoms, and for the most common of the other hydrocarbons present.

Compressibility data for 30 hydrocarbon systems, including 13 gas-condensate fluids, at pressures up to 5,000 lb. per sq. in. in the temperature range 40° to 280° F. are being correlated to provide factors required in measuring gas flowing through high-pressure pipe lines and in estimating underground reserves of gas. The correlation is on the basis of specific gravity of the fluids and liquid:gas ratio at stock-tank pressure. Results obtained with the correlation of the compressibility data indicate that by using a gas-gravity balance and applying conventional liquid:gas ratio measurement procedures in the field, the compressibility of the average natural or gas-condensate fluid can be estimated with an accuracy of 3 percent.

Petroleum-Engineering Studies

Carthage Field, Tex.

A report summarizing the engineering study of the Carthage gas-condensate field in Panola County, Tex., was prepared and has been approved for publication.4/ As an example of the interest manifested in the Carthage field data, the operators in the field, through their Field Engineering Committee, requested and were granted prepublication release of the isopachous maps showing net productive sand thickness of the three major productive zones in the field. Bureau engineers also have been requested by the Field Engineering Committee to furnish large-scale reproductions of the cross sections and correlation sections that were developed during the reservoir studies. These will be supplied upon publication of the report. Because of the extent of the Carthage reservoirs - the field covers over 300,000 acres - operators found it difficult to make individual engineering studies of the whole field. Therefore, the Bureau data are of unusual value to the production engineers in developing and applying modern production procedures with resultant conservation of the reservoir energy and fluids.

New Hope Field, Tex.

The petroleum engineering study of this field, in which water was injected into some of its reservoirs as a stimulating medium early in their life, was published.5/ Because of these pressure-maintenance operations, it is estimated that the ultimate recovery from the field will be doubled. The report has had wide distribution and favorable comment both in this country and in Mexico, where this principle of water injection in the early life of deep-seated reservoirs may have application to the Poza Rica field. In addition to serving industry, the New Hope report is now being used by the University of Texas as a textbook in advanced petroleum-engineering courses.

4/ Wilhelm, C. J., Harris, H. M., Harlin, M. N., Petroleum-Engineering Study of the Carthage Field, Panola County, Tex.: Bureau of Mines Rept. of Investigations 4698, 1950, 60 pp.

5/ Carpenter, Charles B., Anderson, Kenneth F., and Cook, Alton B., Petroleum Engineering Study of the New Hope Oil Field, Franklin County, Tex.: Bureau of Mines Rept. of Investigations 4594, 1949, 88 pp.

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Big Lake Field, Tex.

A petroleum-engineering analysis of the Ellenburger reservoir in the Big Lake field, Reagan County, Tex., was begun in 1949 with the view of publishing a report of the work. This was the first oil-producing dolomitic reservoir discovered (1928) in the fractured Ellenburger formation in West Texas. After producing more than 30,000,000 barrels of oil, it is now the most nearly depleted of the 39 Ellenburger oil-producing reservoirs in the West Texas Permian Basin. Accordingly, the experience gained in this field should be beneficial to persons engaged in the development and operation of other oil-producing reservoirs in this formation. Much interest in the study has been shown by petroleum technologists in industry.

Study of Reservoir-Oil Samples

Elk Basin Field, Wyo. and Mont.

Bureau of Mines engineers completed a series of studies on samples of oil obtained from the bottom of nine wells that produce from the Tensleep sandstone reservoir in the Elk Basin field. These samples were analyzed for such physical characteristics as the volumes of gas in solution, saturation pressures or bubble points, shrinkage in volume caused by the release of gas from solution, expansion of the oil with decrease in pressure, and other related properties. The data from these tests indicate that the oil in the Tensleep reservoir of this field has unusually varying physical characteristics, such as a saturation pressure of 1,250 lbs. per sq. in. absolute and 490 cubic feet of gas in solution in a barrel of oil at the crest of the structure, and a saturation pressure of 530 lbs. per sq. in. absolute and 134 cubic feet of gas in a barrel of oil low on the flanks. Hydrogen sulfide content of the gas in solution in the oil ranges from 18 percent on the crest to 5 percent at wells low on the flanks of the structure. The relations of these and other variable characteristics of the reservoir oil to the position of the oil in the structure are of great interest. These findings were incorporated in a report[6/] that is being published.

Rengely Field, Colo.

The continued study of reservoir-oil samples resulted in a report[7/] on the oil in the Weber sandstone reservoir in the Rangely field. The saturation pressure and solution gas:oil ratio of the oil in that reservoir decreased with depth below the gas-oil contact. The composition of the gas in solution in the original reservoir oil varied with the elevation of the oil in the reservoir. Other characteristics of the oil and gas varied to a much greater degree than might have been expected. Publication of the results will make available to engineers new concepts regarding the possible composition of oil and gas in petroleum reservoirs.

Methods of Estimating Fluid Recovery

In a continuation of the research on oil-reservoir performance through the study of reservoir oil samples, accepted methods of estimating the ultimate recovery from

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6/ Espach, Ralph H., and Fry, Joseph, Variable Characteristics of the Oil in the Tensleep Sandstone Reservoir, Elk Basin Field, Wyo. and Mont.: Bureau of Mines Rept. of Investigations 4768, 1951, 24 pp.

7/ Cupps, Cecil Q., Lipstate, Philip H., Jr., and Fry, Joseph, Variance in Characteristics of the Oil in the Weber Sandstone Reservoir, Rangely Field, Colo.: Bureau of Mines Rept. of Investigations 4761, 1951, 68 pp.

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a reservoir were found to be in error when reservoirs of the solution-gas-drive type contain highly volatile oils. The normal procedure in estimating future reservoir performance requires the use of relative permeability data for gas and oil of the reservoir rock and includes material-balance calculations utilizing data on the volume of the reservoir fluids, oil and gas production with corresponding reservoir pressure decline, and data on the physical properties of the initial reservoir fluids. This procedure permits the estimation of future and ultimate oil and gas production and future gas:oil ratios. The inaccuracy in the procedure results from the false assumption (for reservoirs containing highly volatile oils) that all of the "free" or liberated gas that enters the well bore remains in the gaseous phase as it is produced. However, the volume of the hydrocarbon liquid condensed from the gas from some reservoirs may equal or even exceed the volume of stock-tank oil estimated by present methods.

A method based upon special laboratory analyses of reservoir oil samples, has been developed whereby recognition is given to the additional volume of oil that will be recovered at various stages of depletion from the reservoir. The development of the method resulted from a study of laboratory analyses of reservoir samples and of field data obtained from four reservoirs containing oil ranging from the highly volatile to the relatively low-volatile type.

Well-Spacing Studies

Throughout much of its history the petroleum industry has been confronted with the serious problem of spacing oil and gas wells in an optimum manner. Generally, optimum spacing is construed to be that particular number and distribution of wells, in an oil reservoir, that will yield the greatest profit to the operator consistent with maximum recovery of petroleum. The importance of the problem to the oil industry and to the general public becomes clearly evident when consideration is given to the cost of drilling oil and gas wells, to the significance of crude oil in the national economy, and to the inevitable fact that petroleum is an exhaustible, vital, natural resource.

Although striking progress has been made in recent years in oil-reservoir technology, completely defensible solutions of the well-spacing problem have not been found, largely because of its many inherent and unusual complexities. In this respect, few, if any, oil-field problems have surpassed the well-spacing problem as a source of controversy. Bureau of Mines engineers engaged in well-spacing investigations have concluded that the problem is an inextricable part of the entire realm of oil-reservoir mechanics and that much more must be learned about the interpretation of oil-field data and about the behavior of oil-reservoir fluids (gas, oil, and water) in oil-reservoir rocks before relations of well spacing to oil recovery can be calculated with a high degree of certainty. Engineers in the Bureau of Mines have been investigating the spacing of oil and gas wells by analyzing and correlating oil-field performance data and by making theoretical studies of the behavior of oil-reservoir fluids in porous rocks.

The theoretical study shows that more knowledge of heterogeneous fluid flow must be gained before the well-spacing problem can be reduced to a rational basis of scientific principles. More important, however, it appears that the immediate objective of the well-spacing problem should be to establish completely defensible relationships between well spacing and oil recovery for idealized reservoir systems. Then, a sound basis would be afforded for applying the results in a qualitative manner to oil fields.

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Drilling-Fluid Research

During oil-well coring operations, filtrate from the drilling fluid enters the pore spaces of the cores while they are being cut and partly replaces the originally contained gas, oil, and water. Consequently, if core-analysis results are to lead to reasonably accurate determinations of the oil, gas, and water contents of the undisturbed oil-reservoir rock, it is necessary to consider this filtrate contamination.

Bureau of Mines engineers are continuing their studies to determine the degree to which filtrates from drilling fluids displace the fluids originally contained in cores cut from the formations. A report[8/] of tests on cores cut in wells in the South Coles Levee, Calif., and Rangely, Colo., fields, in the presence of oil-base drilling fluids containing a dissolved organic tracer, has been completed and is being published.

The foregoing tests and tests on cores from a well in the Gulf Coast area indicate that most of the cores contained filtrate from the oil-base drilling fluid, which altered the oil content of the cores but did not affect their water content. Accordingly, this method of coring with oil-base drilling fluid makes it possible to determine more accurately than heretofore the volume of water occurring in petroleum reservoirs. Because the pore space in the cores not filled with water must be filled with oil or gas, the volume of petroleum in the reservoir may be calculated by subtracting the volume of water in the reservoir from the total pore volume of the reservoir.

Some operators believe that the filtrate from drilling fluids reduces the permeability of oil-reservoir sands to oil and thereby restricts the flow of oil into wells. It is not known whether the filtrates alter the physical characteristics of the porous rock formations about the wells, whether the solid particles in drilling fluids plug the pore channels in the rocks, or whether a combination of both actions is prevalent. The properties of the drilling fluid opposite the oil-bearing formation during the period of well completion and during reconditioning is a subject of primary importance, and an investigation has been started to determine the effects of different drilling fluids on the permeability of oil-reservoir rocks for the purpose of obtaining information leading to the preparation of drilling fluids that will cause minimum resistance to flow of crude oil into wells.

Preliminary work has been completed for a series of tests to find out whether it is possible to correlate the water content of reservoir rocks determined by capillary pressure and the water content of a portion of the same rocks determined by analyzing the cores cut with oil-base drilling fluid in the hole. The analysis of the cores has been completed, and the determination of water content by capillary methods is in progress.

A detailed laboratory study is being made of representative samples of a large bentonitic clay deposit in San Benito County, Calif., about 60 miles south of San Francisco. Preliminary results indicate that the clay may meet the requirements of the California petroleum industry for the preparation of high-quality drilling fluids.

8/ Gates, George L., Morris, Frank C., and Caraway, W. Hodge, Effect of Oil-Base Drilling Fluid Filtrate on Analysis of Cores from South Coles Levee, Calif., and Rangely, Colo., Fields: Bureau of Mines Rept. of Investigations 4716, 1950, 25 pp.

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Productivity of High-Pressure Oil and Gas Wells

Research on the productivity of high-pressure oil and gas wells has been directed toward the determination of the pressure loss due to friction in flowing gas wells. To this end, a study, in cooperation with the American Gas Association, has been made of the theoretical background for computing friction factors for flowing gas wells.

The results of the study of the method of computing friction factors were published.9/ Two methods of calculating friction factors were presented. The first method, requiring pressures, temperatures, and specific volumes of the flowing fluids at various depths in the well bore, showed how the mechanical-energy-balance equation for vertical flow may be integrated graphically over the actual pressure-volume-temperature path of the fluid as it moves up the well bore. Thus, assumptions regarding the effective temperature and effective compressibility of the fluid in the well were avoided. The second method presented an equation derived on a basis of the assumptions that both the temperature and the compressibility are fixed at constant effective values throughout the flowing column of gas. The second method provided a convenient and practical means of calculating subsurface pressures in a flowing gas well.

The field testing of a gas well to determine friction factors requires the accurate measurement of subsurface pressures. The general investigation to increase the accuracy of the Amerada-type pressure gage has been carried further by studying the calibration characteristics of six different gages in the pressure range 700 to 7,000 lb. per sq. in. to evaluate the effects of metal hysteresis. A report is in preparation for publication in the near future.

In the field investigation of friction factors for flow in gas wells, data have been collected for two wells with 2-inch tubing, three wells with 2-1/2-inch tubing, three untubed wells with 5-1/2-inch casing, three untubed wells with 7-inch casing, and for the annular spaces between the tubing and casing of four wells. Although much of the information remains to be studied and correlated, a satisfactory solution of the problem of establishing a set of friction factors for gas wells is indicated. With this information available, it will be possible to calculate accurate pressures at the sand face in wells, these data being necessary for determining their productivity.

Sulfur Recovery

In view of the importance of sulfur as a mineral resource, the failure to keep the discovery of new reserves of sulfur in pace with the production of this mineral and the large volumes of hydrogen sulfide associated with a number of domestic oil and gas fields, the sulfur industry has turned to petroleum as an additional source of this important commodity. A Bureau study has indicated that more than 6-1/2 million tons of sulfur can be produced from the hydrogen sulfide present in the oil and gas reserves of Wyoming. These and related data were called to the attention of the sulfur industry at the Sulfur Symposium of the American Chemical Society10/ and are being published.

9/ Smith, R. V., Determining Friction Factors for Measuring Productivity of Gas Wells: Petrol. Trans. Am. Inst. Min. and Met. Eng., vol. 189, Jour. Petrol. Tech. Mar. 1950, pp. 73-82.

10/ Espach, R. H., Sources of Hydrogen Sulfide in Wyoming: Ind. Eng. Chem., vol. 42, 1950, pp. 2235-2237.

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SECONDARY RECOVERY

Field Studies

Humboldt-Chanute Field, Kans.

Case histories of successful water-flooding projects serve as excellent guides for the development and operation of other similar projects. Some of the first water-flooding operations started in Kansas are in the Humboldt-Chanute field in Neosho and Allen Counties, Kans. It is natural, therefore, to turn to this area for experience. The Lynde, Walter, and Darby project in the eastern half of T. 27 S., R. 18 E., was started late in 1937 and is the oldest and largest secondary-recovery project in the field. Because of the continuing expansion of the project, the production from the flood has increased each year since 1944. In 1949 the oil production from the approximately 1,000 acres under flood exceeded 900 barrels daily. On some leases the increased oil recovery by water flooding exceeds 6,000 barrels per acre. Figure 2 shows the production history of this project. A report[11] prepared in cooperation with the Kansas-Oklahoma Water-Flood Operators gives the case histories of the four largest projects. During 1949 these projects produced more than 450,000 barrels of oil from the 1,500 productive acres included.

Weber Pool, Okla.

A report[12] on the petroleum-engineering study of the Weber pool in T. 26 and 27 N., R. 13 E., Washington County, Okla., by Bureau of Mines engineers shows that to January 1, 1950, approximately 14,000,000 barrels of oil had been recovered from the 2,700 productive acres in the field. Secondary-recovery production methods have not been applied intensively in the field, but an analysis of production records indicates that about 3,700,000 barrels of oil was recovered as a result of the application of vacuum and gas drive. Figure 3 shows the production history of the field to the end of 1949. It will be noted that the rate of oil production has been increased gradually for more than 20 years by gas drive. The engineering study shows that the average oil saturation in the reservoir is high and that the field as a whole is well suited for the application of water flooding. It is estimated that the recoverable oil reserve in the field exceeds 17-1/2 million barrels, or nearly 25 percent more than recovered in the 45 years since the field was discovered. Four water-flooding projects have been started in the field recently, but it is too early to estimate the ultimate recovery that will be attained, as none of the projects has yet reached a peak rate of production under flooding.

11/ Joers, J. C., Grandone, Peter, and Taliaferro, D. B., Water Flooding in the Humboldt-Chanute Oil Field, Neosho and Allen Counties, Kans.: Pamphlet, 1950, 19 pp.; also Producers Monthly, vol. 14, No. 8, June 1950, pp. 12-21, 24-25; also Petroleum Engineer (published in 2 parts), part I, vol. 22, No. 6, June 1950, pp. B-11-B-12, B-14, B-16-B-18, part II, vol. 22, No. 9, Aug., 1950, pp. B-56-B-60; also Oil and Gas Journal (published in 4 parts), part I, vol. 49, No. 8, June 29, 1950, pp. 60-62, part II, vol. 49, No. 9, July 6, 1950, pp. 46-48, part III, vol. 49, No. 10, July 13, 1950, pp. 82, 84, 86, part IV, vol. 49, No. 11, July 20, 1950, pp. 91-92, 97-98.

12/ Johnston, Kenneth H., and Moot, C. W., Jr., Petroleum Engineering Study of the Weber Pool, Washington County, Okla.: Bureau of Mines Rept. of Investigations 4740, 1950, 47 pp.

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Brenneman Field, W. Va.

Bureau engineers completed an engineering study of the Brenneman oil field, Hancock County, W. Va., discovered in 1906. The report[13] gives an engineering analysis of the 348-acre field, its development and operation from date of discovery to date of probable abandonment, estimates of the probable total volume of oil to be recovered, and the quantity of oil remaining in the sand. Surface maps of the property and isopachous and contour maps of the sand are presented.

The engineering analysis shows the cumulative oil produced in 17 years of primary-recovery operation amounted to 23.5 percent of the original oil in place; that following a period of abandonment of 3 years, but during both primary recovery and 22 years of secondary-recovery gas-drive operations, 35.0 percent of the original oil was produced; and that with 4 additional years of operation before probable abandonment and using the same production techniques, the total volume of oil recovered would amount to 35.5 percent of the crude oil originally in the reservoir. Furthermore, the oil saturation at estimated abandonment date would be 45.9 percent of total pore volume. The engineering study indicates the possibility of recovering additional oil from the Brenneman field by water flooding.

Oil City - Titusville Area, Pa.

Engineering field studies of three air-gas injection projects in the Oil City-Titusville (Pa.) area reached various stages of completion, and drafting of manuscripts has been initiated. Also, an engineering field study of gas-injection operations in the Cabin Creek field near Charleston, W. Va., was completed and a manuscript written.

Healdton Field, Okla.

Intensive work has been done on the petroleum-engineering study of the 7,500-acre Healdton field, Carter County, Okla., to call attention to the oil reserves in this area that may be recovered by secondary methods. The producing formations in the area are a complex series of lenticular sands, few of which have been flushed by a natural water drive. Gas injection has recovered additional oil on the few projects where it has been applied intensively, but the recent drilling of inside and edge wells has opened new sands to production and added valuable reservoir data. The mineral and liquid content, grain and pore size, permeability, and resistivity have been measured on cores taken during the recent drilling operations to aid in interpreting the reservoir conditions. Samples of produced water have been obtained from different areas and horizons to be used with production data in fixing the extent of natural and accidental water flooding. All data obtained indicate that there are appreciable petroleum reserves in this field that may be recovered by well-engineered water-flooding projects.

History of Water-Flooding in Oklahoma

A survey of water-flooding operations in Oklahoma is nearing completion, and a report on this work is being prepared. This report will present the production history of each of the more than 180 water-flooding projects in the State and will be illustrated with many field maps and production curves.

13/ Tignor, E. M., Nabors, Wm. M., Jennings, Thomas, and Krause, Leon, Secondary Recovery of Oil by Air and Gas Injection in the Brenneman Field, Hancock County, W. Va.: Bureau of Mines Rept. of Investigations 4690, 1950, 26 pp.

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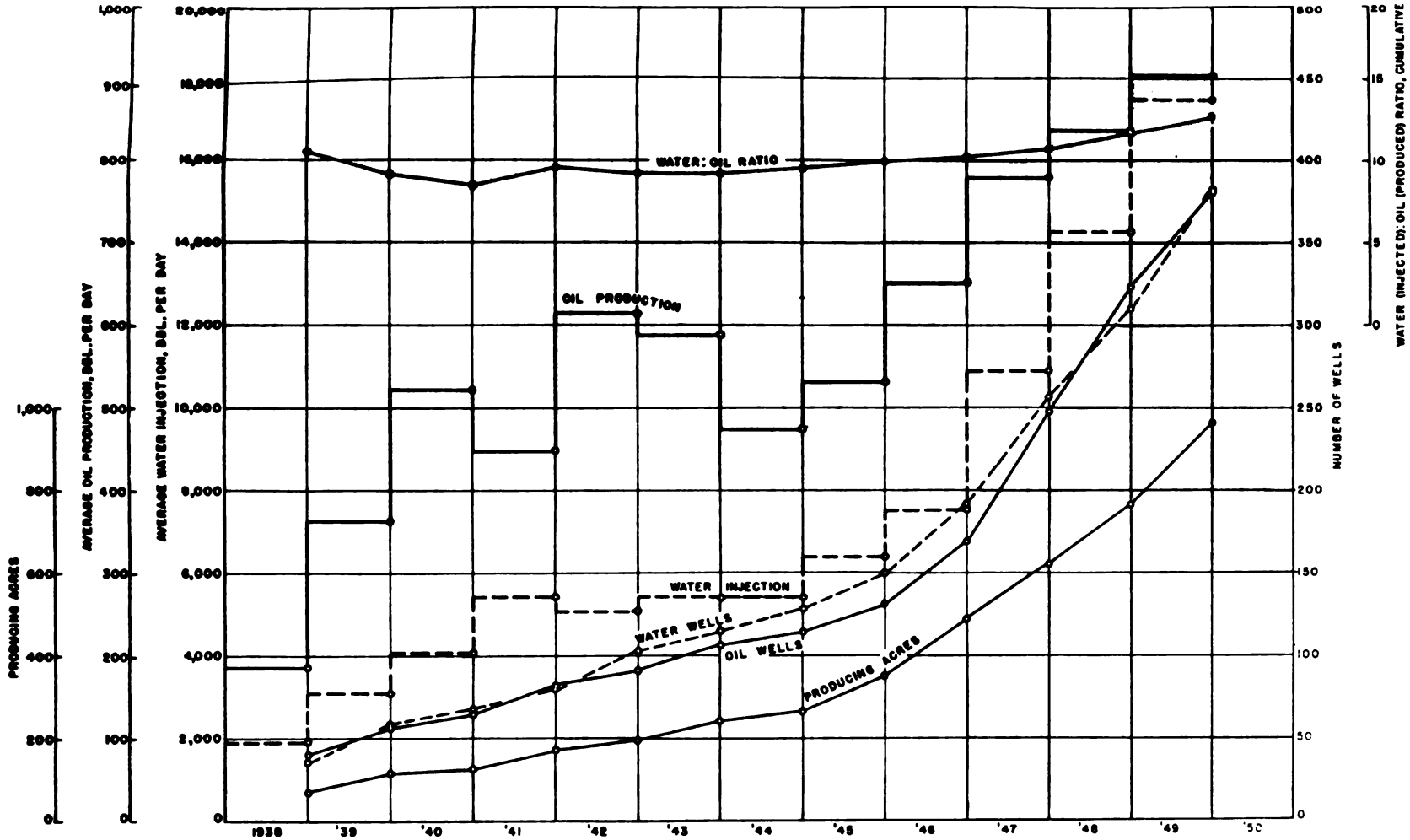

Figure 2. - Production history of Lynde, Walter, and Darby water-flooding project, Humbolt-Chanute field, Neosho County, Kans.

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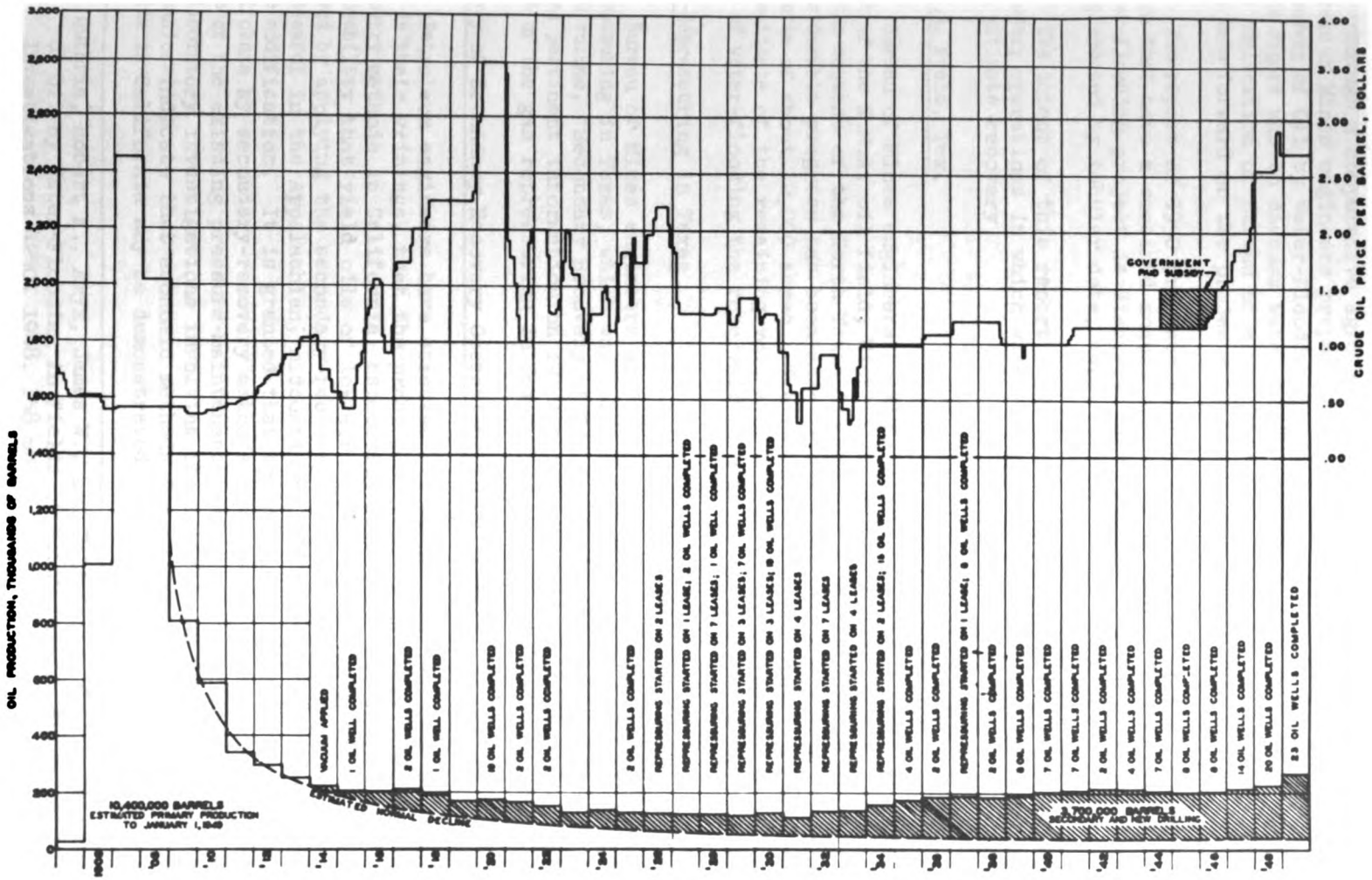

Figure 3. - Oil production by years and the average monthly price paid for a barrel of crude oil, Weber pool, Washington County, Okla.

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Survey of Water-Flooding Projects in North Texas

Through a cooperative agreement with the North Texas Oil and Gas Association, Bureau of Mines engineers prepared a publication entitled "A Survey of Secondary Recovery of Oil by Water-Flooding in the Fields of North Texas." The purpose of this report was to discuss water-flooding projects that have been initiated since the publication of Bureau of Mines Report of Investigations 4250[14/] and to bring the data forward on the projects listed in that report.

The report of 1950 presents discussions of 49 water-flooding projects in North Texas that have a combined annual production of about 550,000 barrels of oil. Each water-flooding project is discussed individually, and the written discussions are supplemented by tabular data, maps, and production-history curves.

The intent of this report is to serve as a guide to present and future secondary-recovery operations in which water-flooding may be an economical method of increasing the ultimate recovery.

K.M.A. Field, Tex.

Bureau of Mines engineers continued the engineering study of the southwestern part of the K.M.A. oil field, Wichita and Archer Counties, Tex., which was initiated as the request of the North Texas Oil and Gas Association's Production Committee. Considerable progress has been made in analyzing the K.M.A. reservoir, which covers an area of about 12,000 acres. The result of this comprehensive analysis will give an estimate of the remaining volume of oil in place and should indicate the feasibility of water-flooding the reservoir as a means on increasing the ultimate recovery.

Gas-Repressuring in Texas

Bureau of Mines engineers assisted in the preparation of a chapter on gas-repressuring in Texas, which was published in the American Petroleum Institute's 1950 volume, "Secondary Recovery of Oil in the United States."[15/] This chapter gives pertinent information on 191 gas-repressuring projects, representing 75 percent of the gas repressuring in Texas.

Survey of Secondary-Recovery Operations in California.

Petroleum engineers have stressed repeatedly, in meetings of technical societies and in their writings, that the problem of recovering additional oil by secondary-recovery methods in California, especially from thick and complex sands of high permeability that yield oils of low A.P.I. gravity and high viscosity, cannot be solved by applying the secondary-recovery methods that have proved economically successful in the Appalachian, midcontinent, and North Texas areas without considerable modification. It is granted that the problem of recovering additional oil in California by secondary-recovery methods once seemed insurmountable, but studies of some of the existing pressure-maintenance and water-flooding projects and the results of laboratory investigations involving theoretical and practical reservoir fluid mechanics indicate that economic methods for recovering additional oil from stripper fields in California may be demonstrated soon.

14/ Guthrie, Robert K., Amyx, James W., and Taliaferro, D. B., Secondary Recovery of Oil by Water-Flooding in Fields of North Texas: Bureau of Mines Rept. of Investigations 4250, 1948, 158 pp.

15/ Gouldy, Roland, and Dobyns, Rollie P., Gas-Repressuring in Texas: Am. Pet. Inst., Secondary Recovery of Oil in the United States, 2d ed., 1950, pp. 592-627.

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A survey of current secondary-recovery operations in California oil fields, including 12 gas-injection projects and 4 water-injection projects, has been made, and a report for publication is nearing completion.

Fields selected for study were those that will best serve to illustrate the principal problems facing secondary-recovery operations in California, to describe the techniques applied to solution of these problems, and to indicate the results thus obtained. It was also desirable to obtain the most representative group of reservoir types and conditions possible. To this end, data were collected, assembled, and analyzed for fields in the San Joaquin Valley, Santa Barbara Coastal District, Ventura District, and Los Angeles Basin. These fields include oil-productive formations typical of Upper Pliocene through Oligocene strata ranging in depths from 1,400 to 11,000 feet subsea and producing various black oils and gas-condensate fluids ranging from 19° to 60° A.P.I. gravity.

Stimulative methods of production include water injection (Richfield and Dominguez fields), waste-water disposal (Del Valle and Greeley fields), pressure restoration by gas injection (Elk Hills and North Belridge fields), and pressure maintenance by gas injection (Kettleman Hills North Dome, Rio Bravo, Greeley, San Miguelito, Newhall-Potrero, Capitan, Canal, North Coles Levee, and West Coyote fields). Although a considerable amount of descriptive information was obtained from the literature, most of the engineering data pertaining to the projects were obtained by direct contact with operating and research personnel in the field.

An essential part of the secondary-recovery studies in California is a series of laboratory experiments, now in preparation, to determine fundamental information needed in the efficient operation of water-flooding projects. One of the most important problems facing water-flooding operators in the State is the effect of oil viscosity on flooding rates. Another pressing problem is the influence of oil viscosity on water:oil ratios at final oil saturations. Apparatus has been designed and is being constructed to determine the nature and magnitude of the foregoing problems over a wide range of oil viscosities.

Special Field Problems

Location of Abandoned Wells

Army mine detectors and M-scope pipe finders have been used effectively to find the general areas of old abandoned wells that may cause operating difficulties, especially in water-flooding and gas-storage projects. Almost invariably, considerable scrap iron is left around abandoned wells, and this junk may be found with the mine detector. The M-scope pipe finder may be used to follow old lead lines that were left in place. More recent work by Bureau of Mines engineers has shown that a dip needle is extremely effective in determining the exact location of wells in which some pipe remains, once the general area has been found with the M-scope pipe finder or the mine detector. Pipe that has remained in the wells for many years becomes highly magnetized and readily attracts the dip needle. Attention now is being directed to methods of finding abandoned wells from which all pipe has been removed. Preliminary experiments indicate that old bore holes will reflect a sound wave created by striking the ground, and that these reflected sound waves may be picked up with a simple geophone. By moving the position of the geophone and observing the change in the intensity of the reflected sound wave it may be possible to locate the old wells. Figure 4 shows the geophones in use in a search for an abandoned well.

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Figure 4. - Geophone being used to pick up reflected sound wave from abandoned well.

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Conditioning Water for Subsurface Injection

The study of plants that condition waters for subsurface injection has been extended to include corrosion tests and chemical analyses at 23 plants in Oklahoma and Kansas, in addition to determining the efficiency of different methods of treating various waters to prevent corrosion of metallic equipment and plugging the sand by precipitated and suspended solids. Repeat tests were made on two plants that had changed from closed to open-type treating systems.

Data now are being tabulated and evaluated as a preliminary to preparation of the final report on the study. These data show that water treatment in some plants is adequate, although in others the conditioned waters are more conducive to corrosion and plugging than the raw waters, which indicates a need for more careful analyses of waters before treating plants are designed and constructed and for more careful control of treatment by plant operators. A preliminary report[16] on this work was presented at a meeting of the American Chemical Society. A summary[17] was published later. Many special chemical tests for the analysis of oil-field brines were developed during the study, and these were made available to the industry in a series of journal articles[18] recently published.

Nonproductive "Black Oil Sands"

A study of the nonproductive black-oil saturated sandstones often found at the base of oil-producing formations in eastern Kansas and Oklahoma is being made on several varieties of the sand found in different fields in that area. Extraction tests were made that show the bitumen content of these samples, on a weight basis, to be of the same order as that of oil produced in a normal manner from other sands. The asphaltene content of the extracted bitumen and the amount of volatile material present did not indicate material of asphaltic consistency in the majority of samples examined. Alteration of the bituminous material during recovery from solutions prevented accurate evaluation of its physical properties, and no appreciable quantity is available from other sources. Insoluble carbonaceous material was found in all samples tested. Clay analyses and the physical properties of the rocks were measured by routine methods. The study disclosed some of the varied characteristics of the black sands but has not offered much encouragement for the recovery of the contained hydrocarbons by economical means.

Selective Plugging of Gas-Injection Wells

Selective plugging of essentially depleted sections of oil sands to prohibit or minimize channeling of injected fluids that otherwise do no useful work in moving oil is one of the most important problems confronting operators of secondary-recovery projects. Great accomplishments have been made in developing products that will plug the sand, but placement of plugging agents in the sand section where needed has not

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16/ Watkins, J. Wade, Arthur, C. E., and Willett, F. R., Jr., Field and Laboratory Studies of Methods of Conditioning Waters for Subsurface Injection: presented 117th meeting, American Chemical Society, Houston, Tex., Mar. 27-30, 1950.

17/ Watkins, J. Wade, Arthur, C. E., and Willett, F. R., Jr., Field and Laboratory Studies of Methods of Conditioning Water for Subsurface Injection: Oil and Gas Jour. vol. 49, No. 15, August 17, 1950, pp. 120, 122, 124, 127-128.

18/ Watkins, J. Wade, Corrosion and Chemical Testing of Waters for Subsurface Injection: Producers Monthly, part I, vol. 14, No. 4, Feb. 1950, pp. 15-19; part II, vol. 14, No. 5, Mar. 1950, pp. 30-31; part III, vol. 14, No. 6, Apr. 1950, pp. 25-31.

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been generally successful. Two products have been used with comparative satisfaction in the Bradford (Pa.) field, but one of these products has not proved useful in the more permeable sands of the midcontinent where water-flooding is practiced. In the Appalachian region, the most urgent need in work on selective plugging is for the development of techniques in applying suspensions of solids in air or liquids in gas-injection wells. Pennsylvania State College and other organizations have done much laboratory work on the plugging of small sandstone specimens with various smokes and liquids, but none of the products has been applied successfully in the field.

The use of solid particles in suspension in water, or chemically treated water, has been investigated in the laboratory, with emphasis on methods of applying them successfully in selectively plugging gas-injection wells. Utilizing especially built laboratory apparatus and sandstone specimens (disks) of cores cut parallel to the bedding plane, one commercially available product was studied intensively, and a technique was developed for introducing the water-suspended solid in gas-injection wells. The physical properties of the suspension were studied. Plugging tests were made with sandstone disks 2.1 inches in diameter and 0.5 inch in thickness and various concentrations of the commercial product. Particle sizes of the suspension were determined, and methods of agglomerating the smaller particles after their penetration into the sandstone were perfected. An optimum concentration of suspended particles was determined. Preliminary tests with sandstones previously invaded with brine indicated the suspension preferentially penetrated the higher permeable sand sections; and the particles that penetrated the lower permeable sand sections, a minute distance under a pressure of 100 p.s.i., could be dissolved by an alkaline wash.

Field tests are in progress in which procedures and information derived during the laboratory study are being applied. An operator of a gas-injection project near Pleasantville, Pa., where gas:oil ratios had increased to a point where the cost of air-gas compression was approaching the net income derived from the sale of the produced oil, set aside a group of wells for testing and the selective plugging of the injection wells. The experiment is being made by Bureau engineers in cooperation with the oil company and the manufacturer of the plugging agent.

Three injection wells and 14 oil-producing wells were included in the original test area. Interesting but inconclusive data were obtained from the injectivity tests made before and after selectively plugging the thief strata (which had formerly conducted the bypassing gas) with a solid-water suspension. The data indicated that three injection wells adjacent to the original pattern disproportionately influenced the producing wells under observation. Additional plugging experiments have been conducted in which the technique employed differed from the original, and the wells are still under observation. Three injection wells and one oil-production well have been added to the testing area.

Selective Plugging of Water-Injection Wells

The completion of the well caliper for use in the selective plugging study has been delayed because of the necessary complexity of the instrument. To permit the use of the caliper in the numerous water-injection wells completed with 2-inch tubing cemented in place, it has been necessary to restrict the instrument to a diameter of 1-3/4 inches. It also has been necessary to provide means of retracting the caliper arms so that the instrument may be withdrawn through the 2-inch tubing. Although these design problems have been difficult, the instrument is now complete, and shop tests are in progress. Field tests will be started soon.

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Relation of Water-Injection Rate to Reservoir Saturation

More accurate information is needed regarding the relationship between the intake capacity of water-intake wells and the liquid saturation of the reservoir. To evaluate this relationship, periodic measurements are being made of the intake capacities of nine input wells on a newly developed 60-acre flooding project in the Bartlesville-Dewey field, Washington County, Okla. Here, water is being injected into the Bartlesville sandstone before any of the producing wells are drilled, thus affording an excellent opportunity to study the behavior of the input wells during the filling of the reservoir.

Electrical Analogy Flow Studies of Gas-Injection and Water-Flooding

An electrolytic model apparatus (see fig. 5) utilizing the basic relationship between Ohm's law of electrical flow and d'Arcy's law of fluid flow through porous media has been employed in the laboratory on engineering field studies of gas-injection and water-flooding projects. The model was utilized to obtain information on well spacing and patterns best adapted to a proposed oil-field water-flooding development in Kansas. A report19/ describing these tests was presented at the 13th Annual Secondary Recovery Conference at Pennsylvania State College.

Studies of Earth Temperatures and Reservoir Oil Samples, Bradford Field, Pa.

Temperature surveys were made in 42 wells dispersed throughout the Bradford oil field, measurements being taken at 100-foot intervals from wellhead to total depth in each well. Where temperature anomalies occurred, and through the oil-sand section in each well, measurements were made at shorter intervals. In the laboratory, seven bottom-hole, crude-oil samples were analyzed. The results of this investigation are being assembled in a report entitled "Earth Temperature and Paraffin Precipitation Studies in the Bradford Oil Field."

Flowing of Oil Wells on Gas-Injection Projects

Work was continued on a field investigation conducted for several years to determine the fundamental characteristics of flow of oil produced from marginal wells on air-gas injection projects through small-diameter, siphon-type flow strings. The initial objective was to determine whether the very small quantities of oil accompanying the injected gas to the producing-well bores on gas-injection projects could be flowed intermittently rather than pumped intermittently, thus utilizing the energy remaining in the injected gas after passage through the sand from injection wells to producing wells to lift the oil to the surface and into tankage. Following some experimentation in the field, the technique proved feasible in wells 300 to 1,100 feet in depth. Flowing of this type has not been tried in deeper wells. After several years of development through laboratory and applied field research, the flowing of marginal wells in the Oil City, Pa., vicinity is considered an improved oil-production technique and is practiced at several places where reservoir conditions permit.

Because flowing permits operating economies not possible with pumping wells, the availability of this technique is of economic importance to operators of gas-injection projects in the middle district of Pennsylvania and perhaps in other oil fields of the Nation.

19/ Eckard, William E., and Mason, Jack A., Electrolytic Model Studies as Applied to Water Flooding a Shoestring Sand: Presented at Thirteenth Annual Secondary Recovery Conference held at Pennsylvania State College, State College, Pa., October 28, 1949 - In Press.

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On July 1, 1950, about 200 oil wells equipped with flow strings designed by Bureau engineers were flowing on 19 leases operated by 5 companies. Of the 200 flowing wells, 150 are being operated on adjacent leases in the Oil City pool, and 81 of these wells are on one lease. During the fiscal year 1950, the only work completed was the collection and analysis of data on the flowing projects under study.

Shooting Oil and Gas Wells with Explosives

In February 1949, a study of oil- and gas-well shooting was begun. A three-fold approach to the problem was started to determine how and why an explosive fractures oil- and gas-bearing rocks, thereby increasing the productivity of shot wells: First, a study to correlate the results of shooting with the physical properties of the rocks affected; second, a survey to determine the physical properties of the oil-bearing rocks obtained from fields throughout the United States where well shooting is practiced, so that any correlations developed by the first study may be applied directly to the shooting of oil and gas wells; third, an analytical study of present oil- and gas-well shooting practices in every producing area and the results as measured in terms of production rates before and after shooting.

The experimental program includes shooting small-diameter holes, drilled to shallow depths in a sandstone sufficiently thick and with suitable lithologic properties, with resistance-type strain gages mounted near the experimental shot hole to determine the strain produced in the rock by the shot. These gages provide a means of judging the extent of fracturing to be expected, whereas injectivity tests on the holes before and after shooting show the increased conductivity resulting from the shot. To test the validity of this approach, and to obtain some data, four series of tests were completed on a lease 3-1/2 miles from Franklin, Pa., where a 50-foot section of the Homewood sandstone (Pottsville group of lower Pennsylvanian age) occurs under a few feet of overburden. These preliminary experiments resulted in two reports.20/

The results of the four series of tests demonstrated that the chosen type of instrumentation met the experimental conditions and provided quantitative data from which the generation and propagation characteristics of the explosion wave could be calculated. As a result of the preliminary work, experimental procedures were developed for the correlation of types of explosives and methods of loading, stemming, and firing the charge, with the strains produced in the rock by the explosion and the results of fluid-injection tests made before and after shooting the test holes.

Additional tests were begun to augment the data obtained in the initial work. Data on (1) a rating of types of explosives in terms of strain waves produced and their generation and propagation characteristics, (2) effect of stemming on performance of different explosives as measured by strain produced, (3) relationships governing the generation and propagation of strain waves from cylindrical charges (also some additional data on spherical charges), (4) time and rate of breakage from an explosion in rock, and (5) correlation of strain data and increase in fluid conductivity of shot wells will be obtained. For these tests, 15 holes 130 to 160 feet

20/ Grant, Bruce F., Duvall, Wilbur I., Obert, Leonard, Rough, R. L., and Atchison, T. C., Research on Shooting Oil and Gas Wells: Am. Pet. Inst. Preprint 826-21-C, presented at the Spring meeting, Eastern District, Division of Production, Cleveland, Ohio, April 27, 1950.

Grant, Bruce F., Duvall, Wilbur I., Obert, Leonard, Rough, R. L., and Atchison, T. C., Use of Explosives in Oil and Gas Wells, 1949 Tests: Bureau of Mines Rept. of Investigations 4714, 1950, 29 pp.

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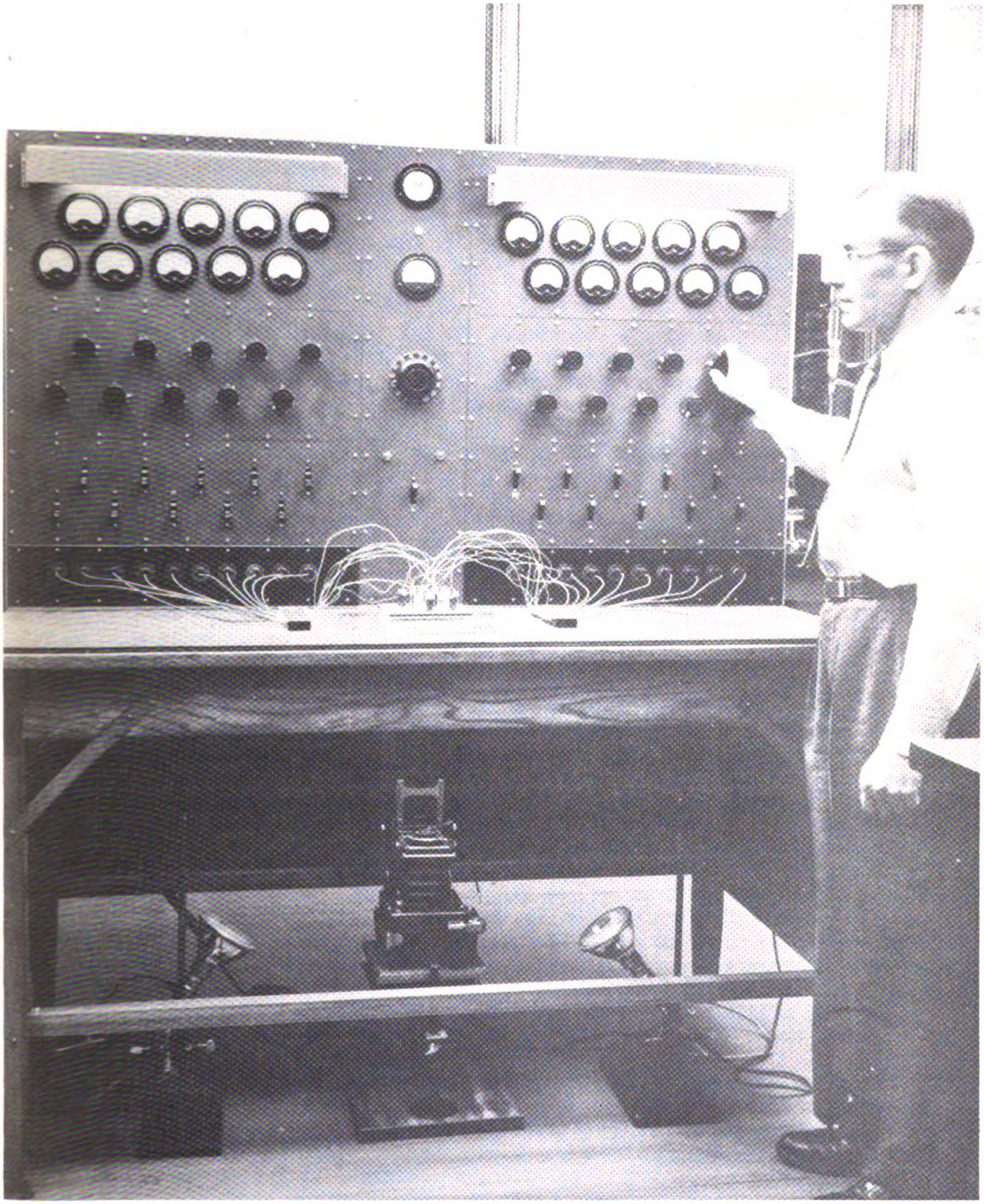

Figure 5. - Electrolytic model used to study well patterns in proposed secondary-recovery operations.

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deep and 148 holes 20 to 30 feet deep were drilled. Most of the 6,205 feet of hole core-drilled was NX (2.95-inch) in diameter. Other hole diameters ranged from BX (2.33-inch) to 8-inch. Everything was in readiness to commence experimental shooting as the year ended.

Activity in the second approach to the problem, a survey to determine the physical properties of oil-bearing rocks from representative fields throughout the Nation, resulted in the acquirement of 126 NX (2-1/8-inch dia.) and larger-diameter rotary cores (about 15 tons) of reservoir rocks from oil and gas fields in the Appalachian, midcontinent, and Rocky Mountain areas. The prompt furnishing of these cores for physical-property testing by a large number of oil and gas companies throughout the Nation is testimony of their eager desire to cooperate in this important part of the study.

Physical-property testing of selected specimens from this valuable library of cores was completed. These tests permit the determination, on diamond-core samples only, of the following rock properties: compressive strength, flexural strength, impact toughness, abrasive and scleroscope hardness, modulus of elasticity, modulus of rigidity, specific damping capacity, and longitudinal bar velocity. In addition, an impact test to determine the breaking strain under high-speed dynamic loading of disks cut from a core sample is being developed, with the expectation that this or similar laboratory tests performed on a core may provide criteria for shot design for the formation or well cored. Although many cores have been furnished by industry, many more will be needed for testing before this phase of the study can be completed.

Laboratory Studies

Core and Water Laboratories

During the year, the core and water laboratory at Bartlesville, Okla., made complete mineral analyses on 265 water samples. A total of 219 core samples were tested for oil and water saturation, porosity, permeability, acid solubility, and connate water by the restored-state method. Numerous miscellaneous analyses were made in connection with various research studies. Much of the work of the laboratory was directed toward the accumulation of a library of analyses of brines produced in Kansas oil fields. The demand for brine analyses has increased greatly in recent months as a result of the greater use of electric logs on a quantitative basis. The resistivities of all brine samples are measured at 60°, 80°, and 100° F.

A laboratory to be used for routine core and water analyses is near completion at Dallas, Tex. This laboratory is relatively small and was designed to take care of the analytical needs of both primary- and secondary-recovery studies conducted by the Dallas and Wichita Falls offices. When not being used in connection with field studies, this laboratory will be used for research.

Oil-formation specimens procured in the Appalachian region from wells drilled on secondary recovery projects and others obtained in connection with special scientific investigations of reservoir characteristics being studied by Bureau engineers are analyzed in the Franklin laboratories. Cores are tested, after preparation with special tools, for porosity, permeability, oil and water saturations, and the chloride content of the contained water.

At Laramie, Wyo., laboratory studies on oil-well cores, oil-field waters, and the clay components of oil-bearing formations developed information on the Newcastle sandstone in the Mush Creek, Fiddler Creek, Skull Creek, and Osage oil fields and on

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the Frontier formation sandstones in the Grass Creek and Salt Creek oil fields in Wyoming. From these and related researches, knowledge was acquired as to how these formations would react to various flooding or driving agents when these are used to recover some of the oil remaining in the formations after primary production.

Correlation of Residual Oil Saturation after Laboratory Air-Drive Tests, with Pore Patterns of Sandstones

Laboratory experiments utilizing air-drive expulsion of oil and water from "fresh" and laboratory-saturated rotary cores were completed. Rotary cores and crude oils representative of many reservoirs were employed in these experiments. Each of 34 flow tests was terminated when the produced air:oil ratio reached values comparable to limiting economic operations in practice. The flow tests were made in flood pots operated in a manner to simulate air-drive secondary-recovery operations in the field. One objective is to learn whether there is a relationship between the volume of oil retained in the sand core following the flood-pot test and the shape, size, and distribution of the sand pores, taking into consideration the viscosity and temperature of the fluids used and the rock characteristics determined by routine core analysis on the pieces of core used in microscopy.

Microscopy studies of pore patterns were conducted to fit this important characteristic of sandstones into an oil-recovery relationship. A method was developed whereby pieces of sandstone, impregnated with a dye-colored plastic, and thin sections, 0.001 inch thick, or polished surfaces are prepared for microscopic examination and color photography.

Owing to the wide interest evidenced in this method of preparing specimens for thin sectioning for microscopy, a report[21] was prepared describing the technique developed.

Color photomicrographs were completed of 152 thin sections representing the 19 radial-sandstone specimens used in the laboratory oil-recovery (oil retained) tests.

The critical dimensions of the pores exposed in one plane, as depicted in the photomicrographs, are being measured, and their significance in relation to the flow experiments is being sought. The year ended with the initiation of the statistical approach to the problem.

Effect of Heat on Oil Recovery

As part of a study of the gravitational drainage of oil from unconsolidated sands, which is being conducted in the San Francisco laboratories, the effect of heat on the caking of oil sands was explored in a series of experiments in which sands of different texture containing oils of different A.P.I. gravities were used. In these experiments, the temperature at the outlet face of a sand-packed tube saturated with oil and water was raised by an electric heating element that was thermostatically controlled.

After drainage of oil from the sand column at room temperature had ceased, the temperature at the outlet face was increased to 120° F., and subsequently by increments of 50° F., until the final temperature of 370° F., was reached. After each temperature increase, oil was permitted to drain from the sand tube until drainage ceased, after which the temperature at the sand face was increased 50° F.

21/ Lockwood, William N., Impregnating Sandstone Specimens with Thermosetting Plastics for Studies of Oil-Bearing Formations: Am. Assoc. Petrol. Geol., Bull. 34, 1950, pp. 2061-2067.

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The results of these experiments indicate that not only is the efficiency of gravity drainage at room temperature dependent on the water content of unconsolidated sands but that, to get enough heat back into sands to reduce materially the viscosity of low A.P.I. gravity residual oils, temperatures at the outlet sand faces must be so high that the sand faces become caked and relatively impervious to the flow of oil through them. Apparently the temperature at which sand saturated with oil, or with oil and water, will cake as a result of heat depends upon the size of the sand grains and the water content of the sands.

Corrosion of Metals by Dissolved Gases in Water

Laboratory apparatus was constructed and assembled at Bartlesville, and tests were started to determine the corrosivity to metals of the dissolved gases, oxygen, free carbon dioxide, and hydrogen sulfide in water, where all other controllable factors influencing corrosion are maintained constant. Tests have been made with dissolved oxygen in the range 9 to 0 p.p.m. in distilled water as the corroding medium, and a significant oxygen-corrosion curve has been obtained. Other tests will be made with various concentrations of free carbon dioxide and of hydrogen sulfide and with different ratios of oxygen to free carbon dioxide and of free carbon dioxide to hydrogen sulfide. The procedures followed are similar to those used in field corrosion tests. The laboratory apparatus is adaptable to and will be used for studies of experimental methods of water conditioning; also, for a study of the effect on corrosion of electric currents caused by bimetallic construction and by changes in fluid velocities.

Tracers in Waters Used for Subsurface Injection

Equipment and facilities are being prepared for work on the development of a suitable radioactive tracer for water injected into oil sands during water-flooding operations. Radiation-detection and monitoring instruments have been obtained, and a radio-chemical laboratory at the Bartlesville Station is now under construction. (See fig. 6.) Initial laboratory work is being started on oil-field cores, using stable ions as tracers. This work will serve as a guide in the selection of radio-isotopes to be used.

Retention of Oil by Reservoir Rock

Surface Forces in Petroleum Reservoirs

The investigation of interfacial-energy phenomena at solid-fluid interfaces has been attacked from three angles: First, a new method had been developed that permits precise measurements of permeabilities of packed beds of sands and powders to flowing liquids. The results of permeability measurements made by the new method, a pressure-decline technique[22] using liquids, agree closely with values calculated from specific surface areas determined by precise, quantitative microscopy techniques perfected as part of this work. Further work has demonstrated that the permeabilities of beds of fine powders to liquids vary with the nature of flowing liquid.

The second phase of the work is concerned with the effect of streaming potentials on the liquid permeabilities of beds of fine powders. A theoretical analysis

22/ Dodd, C. G., Davis, J. W., and Pidgeon, F. D., Measurement of Specific Surface Areas of Nonporous Powders by a Pressure-Decline, Liquid Permeability Method: Presented before Division of Colloid Chemistry, 116th meeting, American Chemical Society, Atlantic City, N. J., Sept. 18-23, 1949.

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of the phenomena involved has been made, and preliminary exploratory studies have been completed. An apparatus is being assembled for use in making precise measurements of streaming potentials. Other work is concerned with gas-adsorption measurements. An apparatus built for making these measurements will make it possible to determine specific surface areas of fine powders and to calculate free energies of adsorption for the solid-liquid systems used in the permeability research.

A study of the types of clay minerals in petroleum reservoir rocks was started. X-ray diffraction procedures have been used to study a number of known reference-clay mineral standards, and these have been applied to the identification of clays separated from petroleum-reservoir rocks. A paper is being prepared that describes an improved color test developed for the identification of clay mineral families when x-ray diffraction, thermal analysis, and other more precise but expensive methods are not available.

For the study of interfacial energy relationships at fluid-fluid interfaces, a pendent drop apparatus is being assembled that will permit precise measurements of the interfacial tensions of systems containing surface-active constituents. A convenient cell-stage[23] for measuring liquid-liquid-gas contact angles was developed.

Film-Forming Substances

Surface-active and film-forming substances have been isolated from oil produced in the Oklahoma City, Okla., and the Rio Bravo, Calif., fields. A report[24] on the extract from the Oklahoma City field crude oil shows that it is composed mostly of normal paraffins of unusually high molecular weight. Further work on the identification of the extracts from both the Oklahoma City and Rio Bravo oils indicate that they contain surface-active constituents of unusual structure.

Recovery of Hydrocarbons from Bituminous Sandstones

In a continuation of the study of the recovery of hydrocarbons from the Edna, Calif., bituminous sandstones, a series of laboratory separation-plant tests on the sandstones from the Vernal and Sunnyside, Utah, deposits showed that these sandstones also are susceptible to the hot-water method of treatment. Furthermore, the tests showed that the bitumen in the Vernal sandstone was extracted more readily by the action of the heated water than was the Edna bitumen, which greatly simplified plant operating procedure. The Vernal sandstone contains no water-soluble iron compounds, such as are found in the Edna sandstone, and therefore requires no preliminary water washing. Recovery of bitumen from the Vernal sandstone was about 96 percent. In contrast to the softness of Edna and Vernal sandstones, the Sunnyside sandstone is an extremely hard asphalt rock that must be crushed to small size to facilitate complete disintegration and displacement of the bitumen from the sand grains in the pulping operation. The disintegrated sand is fine and contains a high percentage of silt that combines to increase the loss of bitumen to the tailings discharged from the plant. Nevertheless, about 90 percent of the original bitumen in the sandstone is recoverable by the hot-water washing process.

23/ Fox, William, A Convenient Cell-Stage for Fluid Profile Measurements: Review of Scientific Instruments, vol. 21, No. 5, May 1950, pp. 499-500.

24/ Denekas, M. O., Carlson, F. T., Moore, J. W., and Dodd, C. G., Isolation and Analysis of Materials Adsorbed at Crude Petroleum-Water Interfaces. I. Normal Paraffins of High Molecular Weight: Presented before Division of Petroleum Chemistry, 117th meeting American Chemical Society, Houston, Tex., Mar. 27-30, 1950.

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Figure 6. - Architect's sketch of entrance to radiochemical laboratory.

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PETROLEUM CHEMISTRY AND REFINING

Ever since the end of World War II, an objective long-range program of petroleum chemistry and refining has been followed, designed to obtain as complete a picture as possible of the availability of fuels for all types of internal-combustion engines, whether they be spark-ignition, compression-ignition, or jet-type engines. As used here, "availability" not only includes supply and production figures but also considerations of quality and utilization characteristics as these factors affect supply. Because of limitations in money and manpower, because knowledge of spark-ignition combustion fuels is much more advanced than that of Diesel and jet fuels, because the use of Diesel and jet fuels is increasing and will continue to increase rapidly, and because there is great need for fundamental data on Diesel and jet fuels, the major emphasis since 1945 has been on fuels in the Diesel-fuel boiling range. The ultimate objectives of these studies of the less volatile fuels could well be stated as the elucidation of the relationships of combustion phenomena of hydrocarbons with their structure and molecular weight and the effect sulfur and nitrogen compounds exert upon these relationships.

The prosecution of this long-range program requires integrated studies of several types, such as surveys of source materials and finished products, improvement of analytical methods, analysis of fuel, and development of suitable methods for studying combustion phenomena. For example, the studies on Diesel fuels currently underway present the following questions, many of which are under investigation:

1. What hydrocarbons or hydrocarbon types are involved, and how can they be determined?

2. What sulfur and nitrogen compounds are involved, and how can they be determined?

3. What combustion phenomena are important, and how can they be studied?

4. What are the combustion characteristics of pure hydrocarbons, and can they be applied to hydrocarbon mixtures?

5. What effect do sulfur and nitrogen compounds have upon the combustion of hydrocarbons?

6. Can physical property and combustion data on pure compounds and their mixtures be translated adequately so as to apply to the combustion of petroleum distillates, and can these data be applied to full-scale engine performance?

In the following discussion, studies or research on most of the above items will be noted, and reference to previous annual reports will show that not only has there been progress in meeting objectives, but that as some have been met the research has been directed toward new goals. An excellent example of such progress is shown by the virtual completion of the development of the constant-volume combustion bomb and its current use in studying combustion phenomena.

To facilitate discussion of the work, it will be considered under six divisions, namely: (1) Crude-oil evaluation, (2) nitrogen in crude oil, (3) sulfur compounds in petroleum, (4) composition of fuels and utilization characteristics, (5) fuel surveys, and (6) composition and properties of asphalts.

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Crude-Oil Evaluation

Routine-Type Analyses

For over 30 years the Bureau of Mines has maintained a file of crude-oil analyses in which are represented all major fields of the United States and Canada and many major fields in other foreign countries. In addition, over 3,000 analyses of smaller fields are included. During the last 10 years, over 1,200 of these analyses have been published in 13 Bureau of Mines reports. These analyses long have been recognized as a source of information regarding the characteristics of crude oil. A recent report[25] presents correlations that enable a person to use the analyses for estimating such properties as aromatic content, sulfur distribution, viscosity relationships, and aniline points of distillates up to 600° F.

Because of the increasing importance of foreign crude-oil production, particularly from Asiatic fields, a report[26] of analyses of oil from the Middle East, South America, and Canada was prepared.

Several reports were issued giving analyses of domestic crude oils. One covered production in the Southeastern States,[27] and 52 analyses of West Texas crude oils were issued to the press in tabular form.[28] This type of release was used to meet the many requests for information regarding analyses of crude oil from the Scurry County area of West Texas, the most active oil-field development in the United States during the past year.

Crude-oil samples were obtained and analyzed from 46 recently discovered fields in Kansas, Oklahoma, California, and Texas that show indications of developing into important fields with respect to crude-oil reserves. A series of reports was inaugurated regarding these crude oils and the significance of their routine analyses, and the first two reports have been published.[29]

Special Analyses

Two oils from Scurry County pool, Tex., were processed to obtain 400° F. end-point gasolines. The gasolines obtained were subjected to usual inspection tests, and the data are included in the report on Scurry County crude oils.[29]

During the current year at the Bartlesville Station, 155 samples of crude oil from 88 domestic fields and 9 from foreign fields were analyzed. The geographical distribution of the samples analyzed was as follows:

25/ McKinney, C. M., Holliman, W. C., and Sponsler, C. R., Composition of Petroleum. Part II. A Correlative Study of Routine and Special Analytical Data and Their Application in the Interpretation of Crude Oil Analyses: Bureau of Mines Tech. Paper 722, 1950, 55 pp., 18 figs.

26/ Blade, O. C., Garton, E. L., and McKinney, C. M., Analyses of Some Crude Oils from the Middle East, South America, and Canada: Bureau of Mines Rept. of Investigations 4657, 1950, 45 pp.

27/ Blade, O. C , and Garton, E. L., Analyses of Crude Oils from Some Fields of Southeastern United States: Bureau of Mines Rept. of Investigations 4687, 1950, 36 pp.

28/ Bureau of Mines, Petroleum Experiment Station; Refinability of West Texas Crude Oils: Oil and Gas Jour., vol. 48, No. 51, April 27, 1950, p. 194.

29/ Smith, N.A.C , Smith, H. M., and McKinney, C. M., Refining Properties of New Crudes. Part I. Significance and Interpretation of Bureau of Mines Routine Crude-Oil Analysis: Petroleum Processing, vol. 5, No. 6, June 1950, p. 608. Part II. Characteristics of Crude Oils from the Scurry County, Tex., Area: Petroleum Processing, vol. 5, No. 7, July 1950, p. 730.

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United States: Alaska 3, California 4, Illinois 2, Kansas 29, Louisiana 6, Oklahoma 10, Pennsylvania 2, Texas 98, and Wyoming 1.

Foreign: Bahrein 1, Canada 3, Chile 1, Columbia 1, Egypt 2, and Iran 1.

Routine-Type Analyses Performed at High Altitude

For many years the Bureau of Mines has maintained a catalog of crude oils consisting of comparable analyses of crude oils from most of the fields of the United States. Until recently, this work was handicapped in the Rocky Mountain region because of failure to obtain comparable analyses owing to the difference in altitude and consequent changes in distillation temperatures. However, the development of pressurized equipment (fig. 7) designed to simulate sea-level pressure has obviated this difficulty. At the Laramie station during the fiscal year 1950, 67 crude oils from the following States have been analyzed: Colorado, 18; Montana, 3; Nebraska, 1; Utah, 2; and Wyoming, 43. A number of these analyses were made in connection with surveys of Missouri River Basin resources. These surveys were designed to furnish information for the Interior Missouri Basin Field Committee in planning the Missouri River Basin Development Program.

Nitrogen in Crude Oils

Nitrogen in crude oils has been assuming more importance recently as knowledge of its detrimental effect in catalytic cracking processes and its gum-forming potential became available. The Bureau of Mines routine analysis procedure for crude oils does not include determination of nitrogen content. The question as to whether properties now determined, could be used to estimate nitrogen content was investigated.

The nitrogen content of 153 crude petroleums from 17 States and 3 foreign countries was determined. These were selected to give as wide a variety of oils as possible. Experiments on a number of the oils showed that the nitrogen was concentrated in the residuum obtained in the routine analysis. Consequently, it was expected that some property of the residuum, such as carbon residue, would correlate best with nitrogen content. It was found possible to correlate carbon residue with nitrogen content for oils produced from a given geologic system (fig. 8). Calculations of nitrogen content by using such correlations show an average deviation of approximately 20 percent. A paper presenting these data is scheduled for presentation at the September 1950 meeting of the American Chemical Society and subsequent publication.

Crude-oil analyses are frequently compiled for publication. A report describing crude oils from recently discovered fields in the Rocky Mountain region was published 30/ as part of a series describing oils from various parts of the country.

Sulfur Compounds in Petroleum

Work under American Petroleum Institute Research Project 48A on "The Production. Isolation, and Purification of Sulfur Compounds and Measurement of Their Properties" has continued.

The research can be separated into three independent but related phases, which are:

30/ Ball, John S., Wenger, W. J., and Whisman, M. L., Refining Properties of New Crudes: Petroleum Processing, vol. 5, No. 8, Aug. 1950, pp. 842-846.

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1. Synthesis, purification, and determination of properties of sulfur compounds.

2. Determination of thermodynamic data of sulfur compounds.

3. Separation, isolation, and identification of sulfur compounds in crude oils.

The first of these phases is being carried out at Laramie, Wyo., and is discussed below. The second phase is under way at Bartlesville, Okla., and is discussed in a following section on thermodynamics of petroleum. The third phase is being conducted at Bartlesville and is discussed immediately following the description of the work at Laramie.

Synthesis, Purification, and Determination of Properties

The compilation of data on sulfur compounds available in the literature has continued. A report[31] published during the year gives viscosities, surface tensions, refractive indexes, and various calculated functions for sulfur compounds containing up to 14 carbon atoms. The compilation of the properties is being continued to give a comprehensive review, which probably will be published within the next 2 years.

The purification program has resulted in the addition of four purified sulfur compounds to the previous seven. The new compounds are 3,4-dithiahexane (diethyl disulfide), thiacyclopentane, thiacyclobutane, and 2-propanethiol. Each of these compounds has a purity greater than 99.90 mol percent.

These high-purity compounds are used to determine accurate physical and thermodynamic properties, and portions are set aside for distribution as calibration standards. Among the properties determined at the Laramie station are freezing points, densities, refractive indexes, surface tensions, viscosities, and mass, infrared, and ultraviolet spectra. The thermodynamic properties are determined at the Bartlesville station. Through cooperation with the National Bureau of Standards, eight of the purified samples have been made available as standard samples for the calibration of analytical instruments such as spectrometers.

Analytical methods. - One of the purposes of this research is the development of analytical methods suitable for the determination of sulfur compounds or groups of sulfur compounds. To accumulate background for possible methods based on spectroscopic determinations, a program has been started to determine spectral patterns for a large group of sulfur compounds. The compounds are obtained, by purchase or donation, in quantities of approximately 200 ml. The purity of each compound is determined, and it is then distilled to enhance the purity if that step seems promising. The samples are then sent to various cooperating laboratories, mostly those of oil companies, for determination of spectra. Of 42 samples contributed to the program thus far, 28 are in process of spectra determination.

As part of the program of developing analytical methods, analyses of distillates from a number of high-sulfur crude oils have been made by existing methods for group

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31/ Helm, R. Vernon, Haines, William E., and Ball, John S., Sulfur in Petroleum. III. Selected Properties of Some Sulfur Compounds: Bureau of Mines Rept. of Investigations 4566, 1949, 44 pp.

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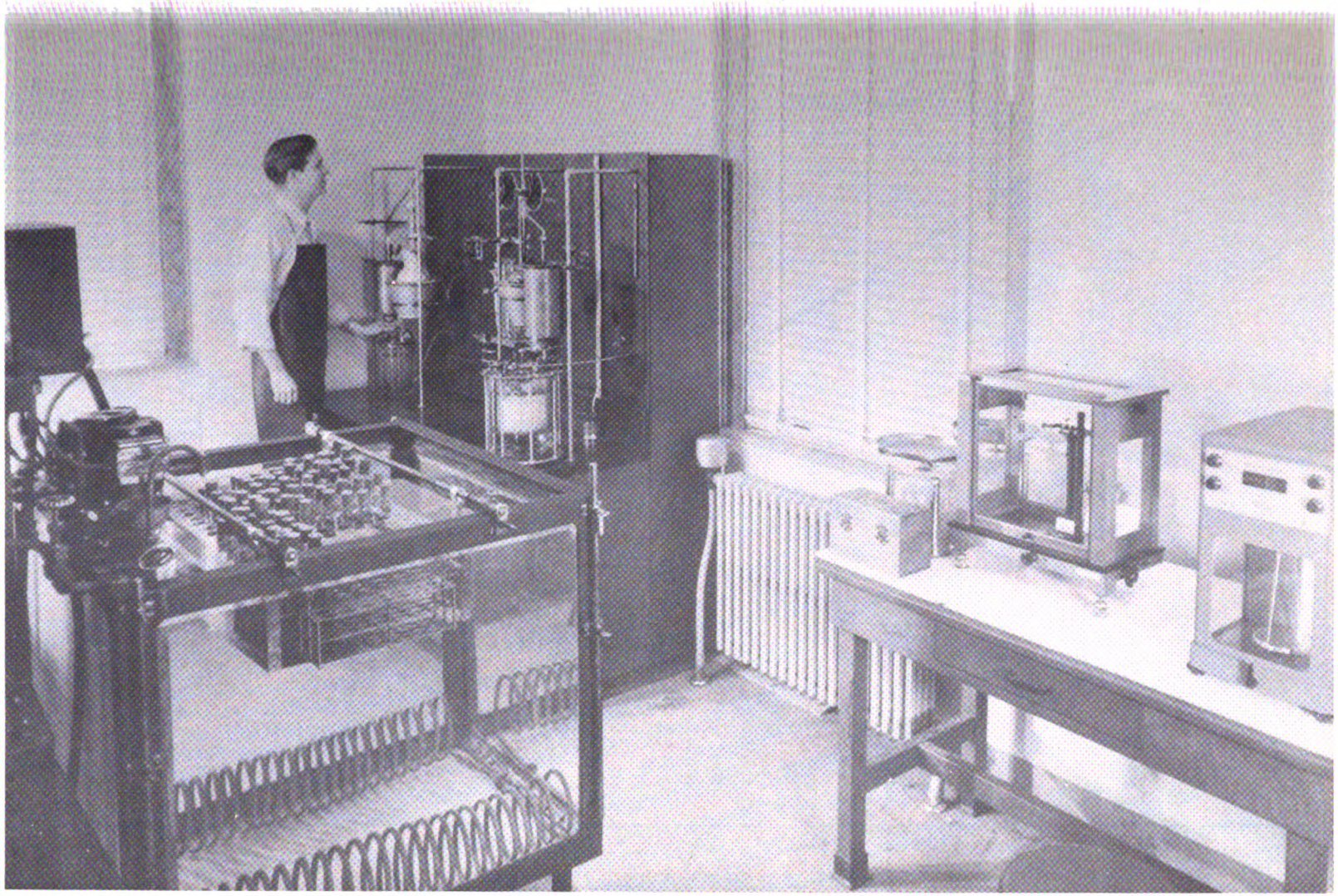

Figure 7. - Laboratory for crude oil analysis showing equipment for pressurizing to sea-level pressure.

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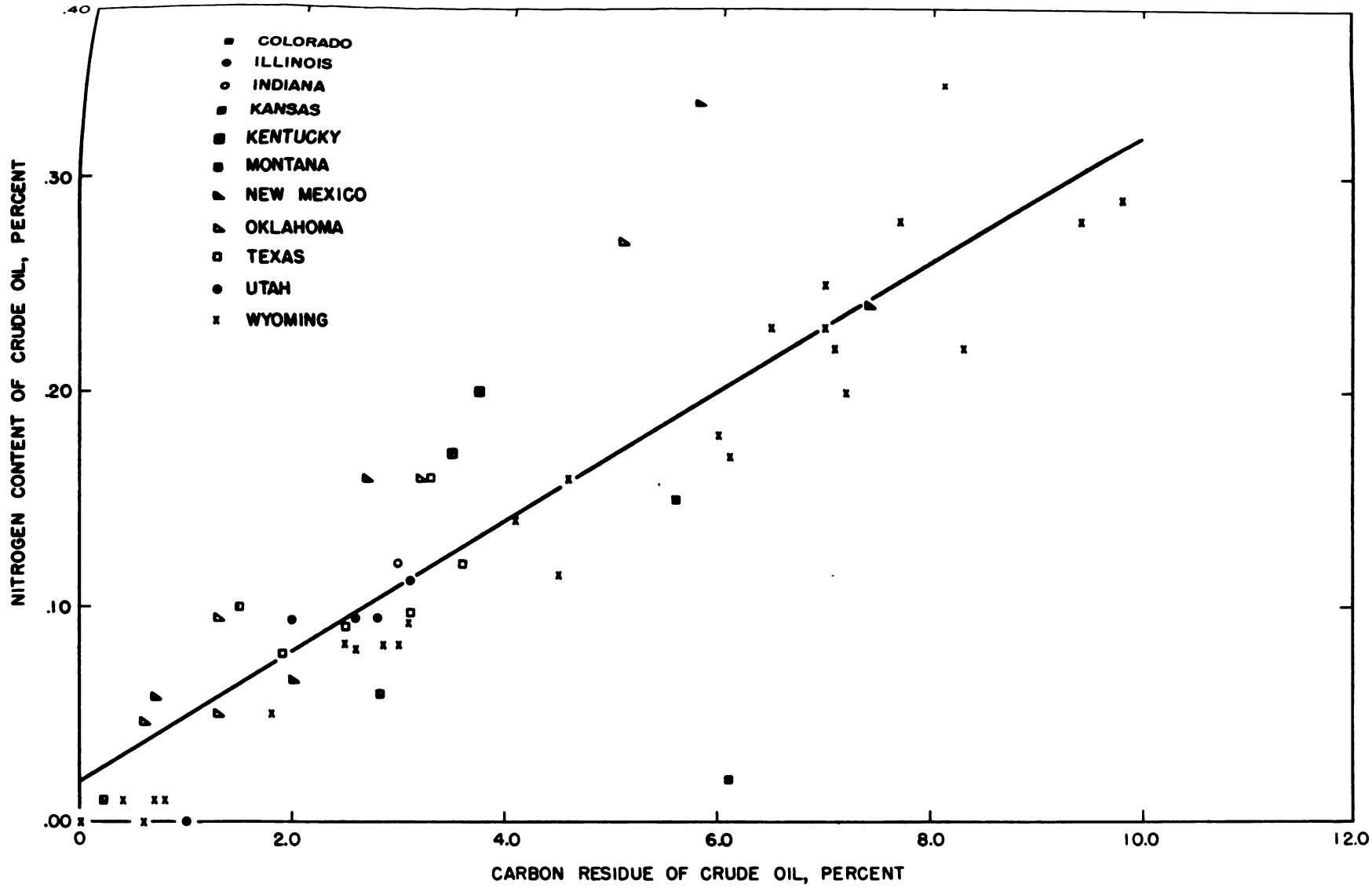

Figure 8. - Relationship of carbon residue and nitrogen content of crude oils produced from formations of Carboniferous system.

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sulfur analysis. These analyses[32] indicate that the sulfur compounds in various crude oils may be considerably different with respect to the type of sulfur linkage. Some of the oils analyzed contained predominantly thiols (mercaptans) and disulfides, whereas others contained chiefly sulfides.

Thermal decomposition of sulfur compounds. - Thermal treatment is the most common operation in petroleum refining. Consequently, it is important to know the reactions that sulfur compounds undergo when they are heated. Such knowledge can be used in designing processes to remove the sulfur when it is in the form most amenable to treatment. Several types of pure sulfur compounds are to be studied at various temperatures and times of exposure to trace the decomposition reactions they undergo.

Apparatus has been designed (fig. 9) consisting of a feed mechanism, a controlled-temperature furnace, and an absorption system. The feed mechanism consists of a hypodermic syringe activated by a positive drive whose speed can be varied. The furnace is a combustion tube with a 560 mm. heated section controlled by a 3-point controller to ± 5° C. throughout its length. The absorbing system will vary for different compounds, but for a thiol it consisted of slant-tube absorbers filled with caustic, followed by a trap cooled with dry ice and carbon tetrachloride. The slant-tube absorbers are used to reduce the head of pressure necessary to put the compound through the system.

The first compound to be studied was 2-methyl-2-propanethiol (t-butyl mercaptan). Temperatures of 300°, 350°, 400°, 450°, and 500° C., with residence times of 10 to 120 seconds, resulted in the observation of no decomposition in some cases to as much as 97 percent in others. The primary reaction is decomposition of the thiol into hydrogen sulfide and isobutene. Subsequent reactions give free sulfur and isobutane.

Separation and Identification

During 1949, Project 48A completed the first survey ever made of the types of sulfur compounds present in virgin naphthas from a variety of high-sulfur crude oils. The data,[32] shown in part in figure 10, indicate a wide variation in the proportions in which the different sulfur types are present. Such data should eventually be helpful in developing proper refining processes for high-sulfur crude oils.

As a result of this survey, Wasson, Tex., crude oil was selected as the first oil for extensive study to identify the individual sulfur compounds and to determine their percentages in the crude oil. To this end, special distillation apparatus was constructed to separate the light distillate from the crude oil with a minimum of thermal decomposition. The equipment is constructed entirely of glass and operates isothermally, steam at atmospheric pressure being used as the heating medium. With distillate from this apparatus available, attempts have been made to concentrate the sulfur compounds in the 0° to 100° C. boiling range by means of adsorption on silica gel. This technique has met with considerable success, as indicated in figure 11, where it can be shown that more than half of the sulfur compounds in the naphtha have been concentrated in a fraction constituting only 1.2 percent of the naphtha. Recent data indicate that alumina may be a more efficient adsorbent for concentrating sulfur compounds in petroleum naphthas.

32/ Hale, J. H., Thompson, C. J., Barker, M. G , Smith, H. M., and Ball, J. S., Distribution and Types of Sulfur Compounds in Straight-Run Naphthas: Presented before Division of Petroleum Chemistry, American Chemical Society, Houston, Tex., March 29, 1950.

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To determine the temperature stability of sulfur compounds occurring naturally in crude petroleum, various crude oils have been subjected to prolonged heating at each of several successively higher temperature levels, and the hydrogen sulfide and mercaptans evolved at each temperature level have been measured. Hydrogen sulfide and mercaptans are probably predominant among the final decomposition products from the thermal cracking of the original sulfur compounds and should therefore be a measure of the extent or ease of decomposition. Figure 12 gives such data obtained with three crude oils.

In the survey mentioned above, two crude oils yielded distillates that contained elemental sulfur. Further experiments have proved that under the low-temperature distillation conditions employed, if free or elemental sulfur is present in a crude oil it will also occur in the distillate from that oil. Further, at high temperature, evidence at hand appears to indicate that the reaction between elemental sulfur and the hydrocarbons can contribute a large part of the hydrogen sulfide appearing at the temperature levels in the vicinity of 300° to 400° F.

Results of earlier studies on desulfurization of crude oil were published.33/

Composition of Fuels and Utilization Characteristics

Diesel Fuel

The availability of Diesel fuel during the past year was appreciably increased, but petroleum refiners and engine builders continued to show an active interest in the over-all Diesel-fuel supply program. The emphasis has shifted somewhat from improving the supply to improving the quality and gaining a better understanding of the fuel characteristics that affect better engine performance.

Composition and analysis. - The Bureau has long recognized that one of the basic problems in Diesel-fuel studies is the relationship between the composition of the fuel and its behavior in the engine. To this end, the Bureau for several years has maintained formal and informal agreements with several organizations to supply composition data on the fuels on which engine studies are being made. For the last several years these industry-government relationships have culminated in annual "Diesel Fuel Conferences" held at the Petroleum Experiment Station, Bartlesville, Okla. At these conferences the petroleum and automotive industry representatives outline the problems that need study, and the Bureau staff reports on the program of its various researches relating to Diesel fuel. These reports will eventually be published as parts of papers on various phases of the research.

The Western Petroleum Refiners Association (W.P.R.A.) has cooperated informally in connection with studies of Diesel fuels prepared from several selected crude oils of different types that are representative of large production. The first fuel to be studied was prepared from West Edmond, Okla., crude oil, a representative midcontinent oil. The Diesel fuel was fractionated into 134 fractions, the aromatics were separated from each fraction by silica-gel adsorption, and the cetane numbers of the original fractions and the paraffin-naphthene portions of the fractions were determined. From other physical properties, the composition in terms of type hydrocarbons, was estimated for the paraffin-naphthene portion and the aromatic portion. Properties were determined on the paraffin-naphthene portion of each fraction to permit the calculation of the average number of rings per molecule and the average molecular weight of the paraffin-naphthene portion.

33/ Hale, J. H., Simmons, M. C., and Whisenhunt, F. P., Catalytic Desulfurization of Crude Oil: Ind. Eng. Chem., vol. 41, 1949, pp. 2702-2708.

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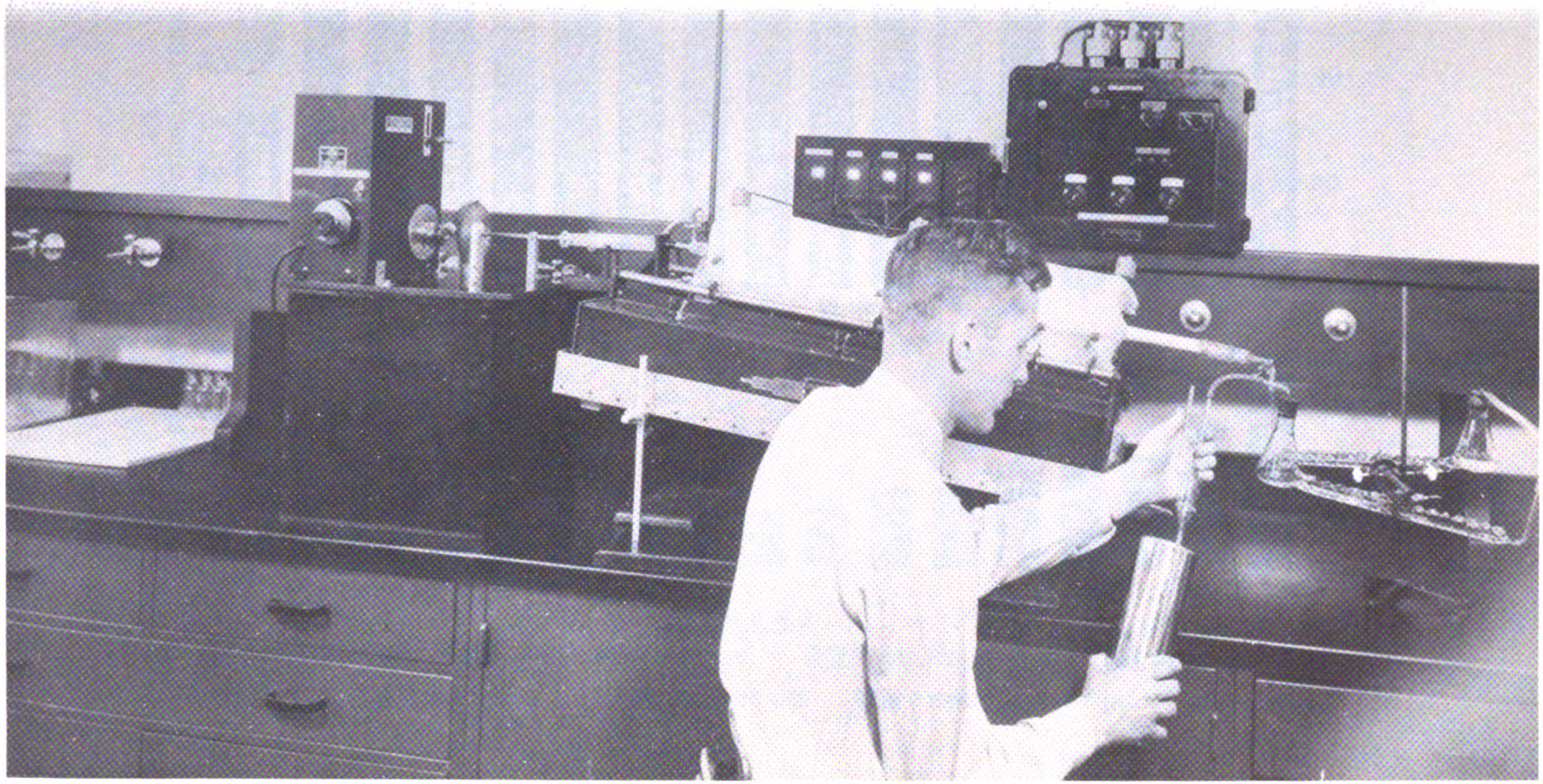

Figure 9. - Equipment for thermally decomposing sulfur compounds.

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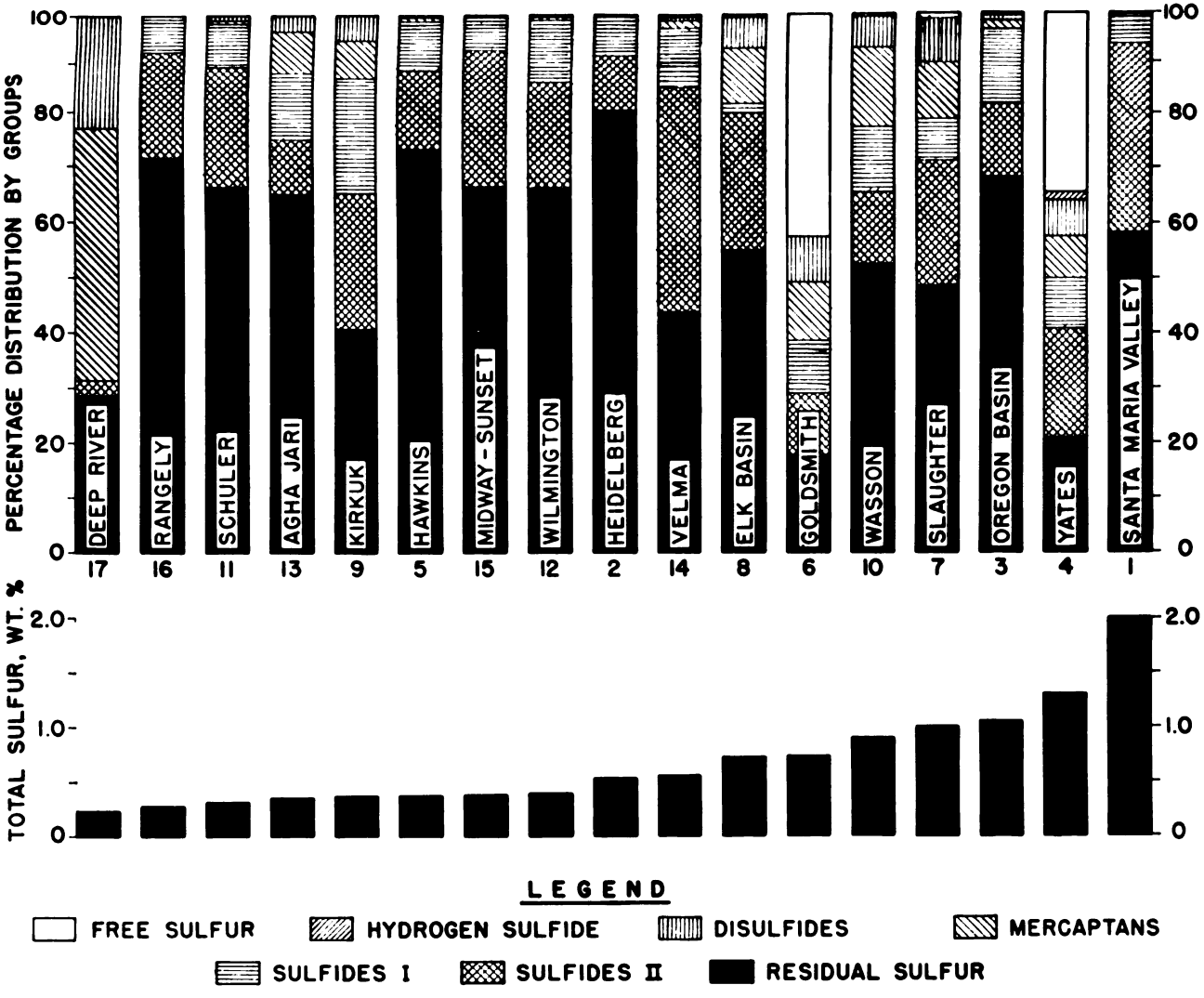

Figure 10. - Group sulfur analyses of naphthas from vacuum distillations.

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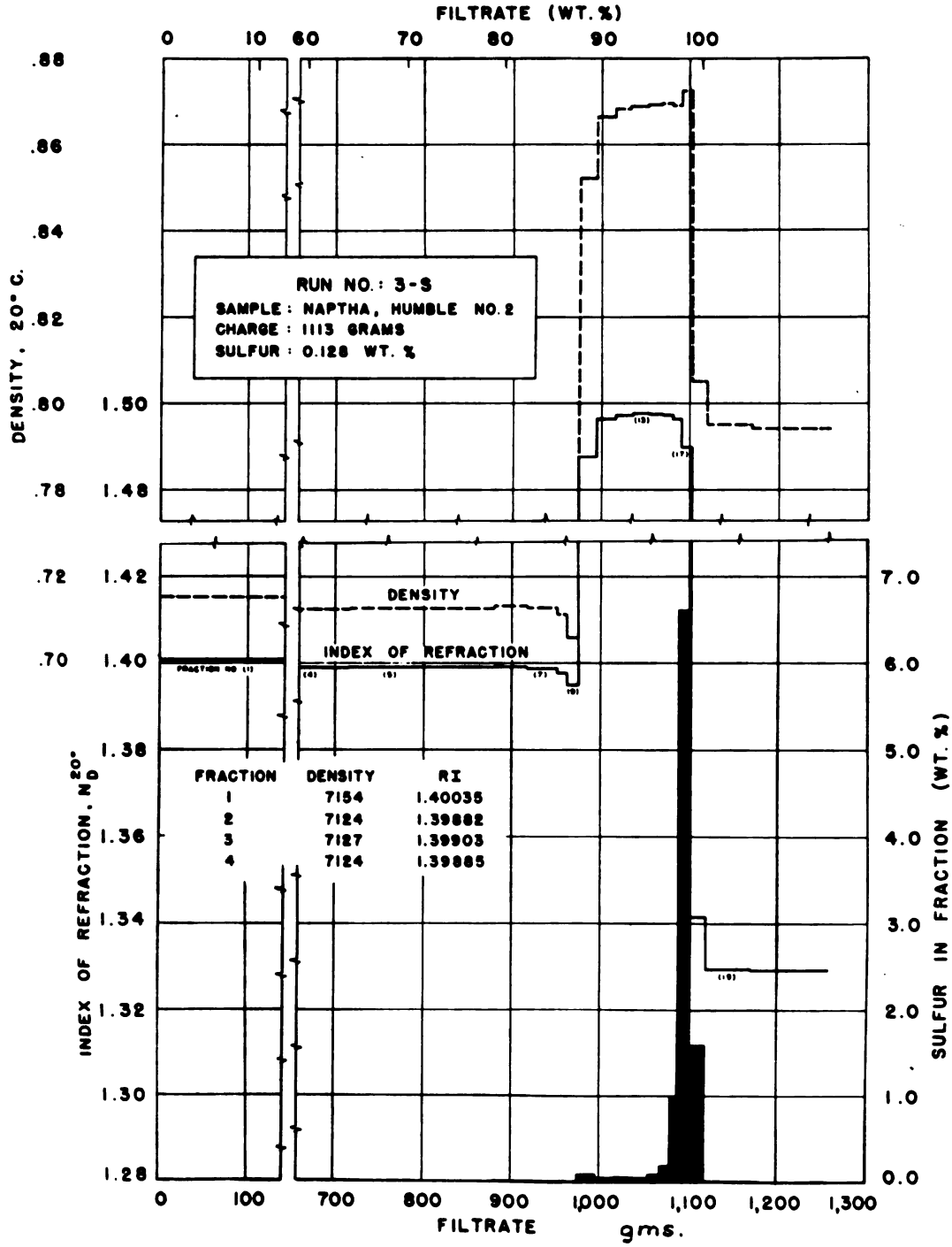

Figure 11. - Silica gel absorptogram of sulfur-containing naphtha.

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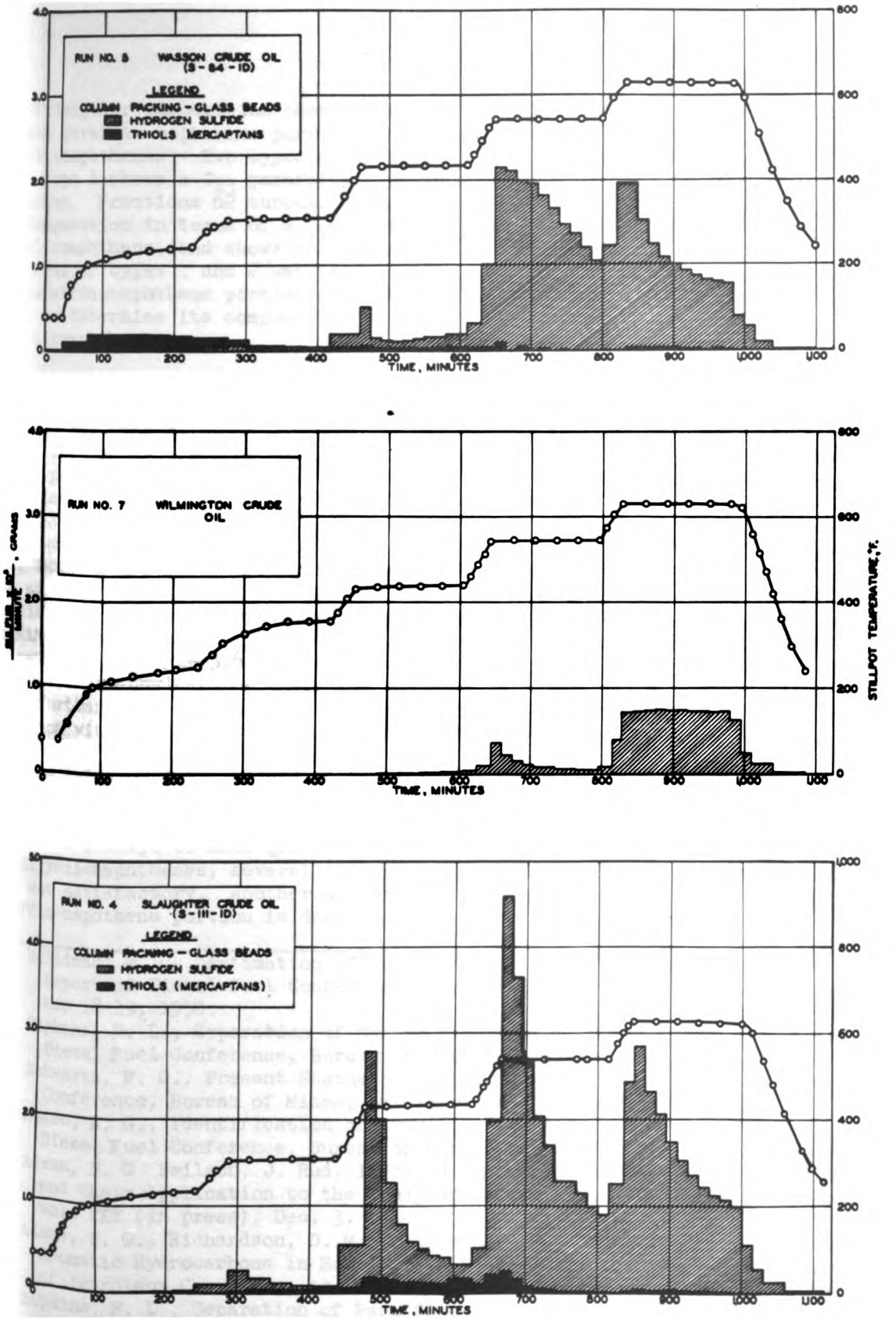

Figure 12. - Sulfur stability tests of selected crude oils.

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Triangular composition charts were then prepared for estimating the composition of each paraffin-naphthene portion in terms of paraffins, monocyclicnaphthenes, and dicyclicnaphthenes. Two types of triangular charts were used, as shown in figure 13. Chart type 1 shows a C_{13} paraffin, a C_{13} monocyclicnaphthene, and a C_{12} dicyclicnaphthene. Fractions 62 through 66 are plotted on this chart. Chart type 2 shows the composition in terms of a C_{14} paraffin, a C_{13} monocyclicnaphthene, and a C_{13} dicyclicnaphthene, and shows the composition of fractions 66 through 75. A series of charts of types 1 and 2 was prepared to cover the boiling range 350° to 650° F. The paraffin-naphthene portion of each fraction then was plotted on the appropriate chart to determine its composition in terms of paraffins, monocyclicnaphthenes, and dicyclicnaphthenes.

A determination of the total paraffin percentage was made on selected fractions by the urea-adduct phenomenon, which makes it possible to separate straight-chain hydrocarbons from mixtures with analogous branched and cyclic compounds.[34] The percentages of n-paraffins, as determined by this method and by the crystallization in the presence of methylal,[35] agreed very well. The estimated composition of the paraffin-naphthene portion of the West Edmond crude oil is summarized in figure 14.[36]

The aromatic portions from 61 consecutive fractions from the West Edmond Diesel fuel, boiling above 400° F., were examined in terms of their ultraviolet spectra. From an examination of these spectra, the following aromatic hydrocarbons have been identified: Tetralin, naphthalene, alpha- and beta-methylnaphthalene, diphenyl, and 2,6-dimethylnaphthalene. In addition, the presence of the following compounds is indicated: 2-methyl—5,6,7,8-tetrahydronaphthalene, betaethylnaphthalene, and 1,6- and/or 1,7-dimethylnaphthalene. The presence of these latter compounds was investigated further by using infrared spectra, selective adsorption techniques, and compound formation with such agents as picric acid and trinitrobenzene.[37]

The value of infrared and ultraviolet spectra as used in hydrocarbon analysis was discussed in papers by Adams and coauthors.[38][39]

In an attempt to make quantitative separation of paraffins, monocyclicnaphthenes, and dicyclicnaphthenes, several different types of adsorbents were used, but none of them was satisfactory. Another analytical tool that is being developed to study this paraffin-naphthene portion is dehydrogenation of the cyclohexanes to aromatics.[40]

34/ Holliman, W. C., Estimation of Normal Paraffins by the Urea Adduct Method: Report of Diesel Fuel Conference, Bureau of Mines, Bartlesville, Okla., May 18-19, 1950.

35/ Hopkins, R. L., Separation of Normal Paraffins by Crystallization: Report of Diesel Fuel Conference, Bureau of Mines, Bartlesville, Okla., May 18-19, 1950.

36/ Schwartz, F. G., Present Status of Composition Studies: Report of Diesel Fuel Conference, Bureau of Mines, Bartlesville, Okla., May 18-19, 1950.

37/ Adams, N. G., Identification of Aromatics by Ultraviolet Spectra: Report of Diesel Fuel Conference, Bureau of Mines, Bartlesville, Okla., May 18-19, 1950.

38/ Adams, N. G. Neilson, J. Rud, Infrared Adsorption Spectra of Six C_{14} Hydrocarbons and their Application to the Study of Diesel Fuel: Proceed. Okla. Acad. Sci., vol. XXX (in press), Dec. 3, 1949.

39/ Adams, N. G., Richardson, D. M., Ultraviolet Spectrometric Identification of Aromatic Hydrocarbons in Some Diesel Fuel Fractions: Presented before Division of Petroleum Chemistry, Am. Chem. Soc., Houston, Tex., March 28, 1950.

40/ Hopkins, R. L , Separation of Paraffins, Monocyclic Paraffins, and Bicyclic Paraffins by Adsorption and Dehydrogenation: Report of Diesel Fuel Conference, Bureau of Mines, Bartlesville, Okla., May 18-19, 1950.

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By this means it is hoped that most of the cyclohexanes can be dehydrogenated to the corresponding aromatics, which can be removed by adsorption or identified by ultraviolet spectra.

A paper[41] was presented before the Western Petroleum Refiners Association giving some of the results of the cooperative work with that organization.

The Bureau has analyzed a Fischer-Tropsch Diesel fuel and a catalytically cracked Diesel fuel as a part of the Navy's extensive program on Diesel fuels as related to future sources of supply The analysis of the Fischer-Tropsch fuel has been completed, and it was reported in a paper[42] presented before the American Chemical Society.

The catalytically cracked fuel was analyzed, using the same general procedure as used for the West Edmond Diesel fuel. The composition in terms of type hydrocarbons, as determined by using the triangular charts mentioned above, is shown in figure 15. The catalytically cracked fuel had a cetane number of 29, as compared to 56 for the West Edmond Diesel fuel. The reason for this difference is readily apparent from an examination of the composition of the paraffin-naphthene portions. The paraffin-type hydrocarbons have high cetane numbers, the monocyclicnaphthenes intermediate, and the dicyclicnaphthenes low. The composition diagrams indicate that the West Edmond fuel has a high percentage of paraffins and very few dicyclicnaphthenes, whereas the catalytically cracked fuel has a high percentage of dicyclicnaphthenes and relatively small percentages of paraffins.

A paper describing a pilot-plant size continuous still used in preparing naphthas and Diesel fuels from crude oils was published.[43]

The American Society for Testing Materials (A.S.T.M.) has conducted cooperative work on the development of a silica-gel method for analyzing Diesel fuels; also methods for determining refractive indexes to the fifth decimal place and for determining density to the fifth decimal place. The Bureau was an active cooperator in this work, and the resulting methods will appear as "publication for information only" in the 1950 A.S.T.M. Standards on Petroleum Products.

A sample of cyclooctatetraene was purified, and its physical properties and infrared and ultraviolet spectra were determined.[44]

Utilization. - Complementing the researches on sources, composition, and properties of Diesel fuels, researches on utilization were directed more toward combustion research of Diesel-fuel fractions and of pure hydrocarbons in the range C_{10} to C_{20}, as described under following headings. Major emphasis was placed on studies probing the fundamental behavior of different type fuels during the combustion process and the manner in which that behavior is affected by changes in the conditions under which the combustion occurs.

41/ Ward, C. C., Relationships of Composition and Ignition Quality of Diesel Fuel: Presented before Diesel Fuel Clinic, Western Petroleum Refiners Association, Alma, Mich., Aug. 19, 1949.

42/ Ward, C. C., Schwartz, F. G., and Adams, N. G., Composition of Fischer-Tropsch Diesel Fuel (Cobalt Catalyst): Presented before Petroleum Division, Am. Chem. Soc., Houston, Tex., March 28, 1950.

43/ Ward, C. C., and Schwartz, F. G., Construction and Operation of the Pilot-size Continuous Still: Petroleum Processing, vol. 5, No. 2, Feb. 1950, p. 164.

44/ Eccleston, B. H., Coleman, H. J., and Adams, N. G., The Purification and Some Physical Properties of Cyclooctatetraene: Jour. Am. Chem. Soc., vol. 72, 1950, pp. 3866-3870.

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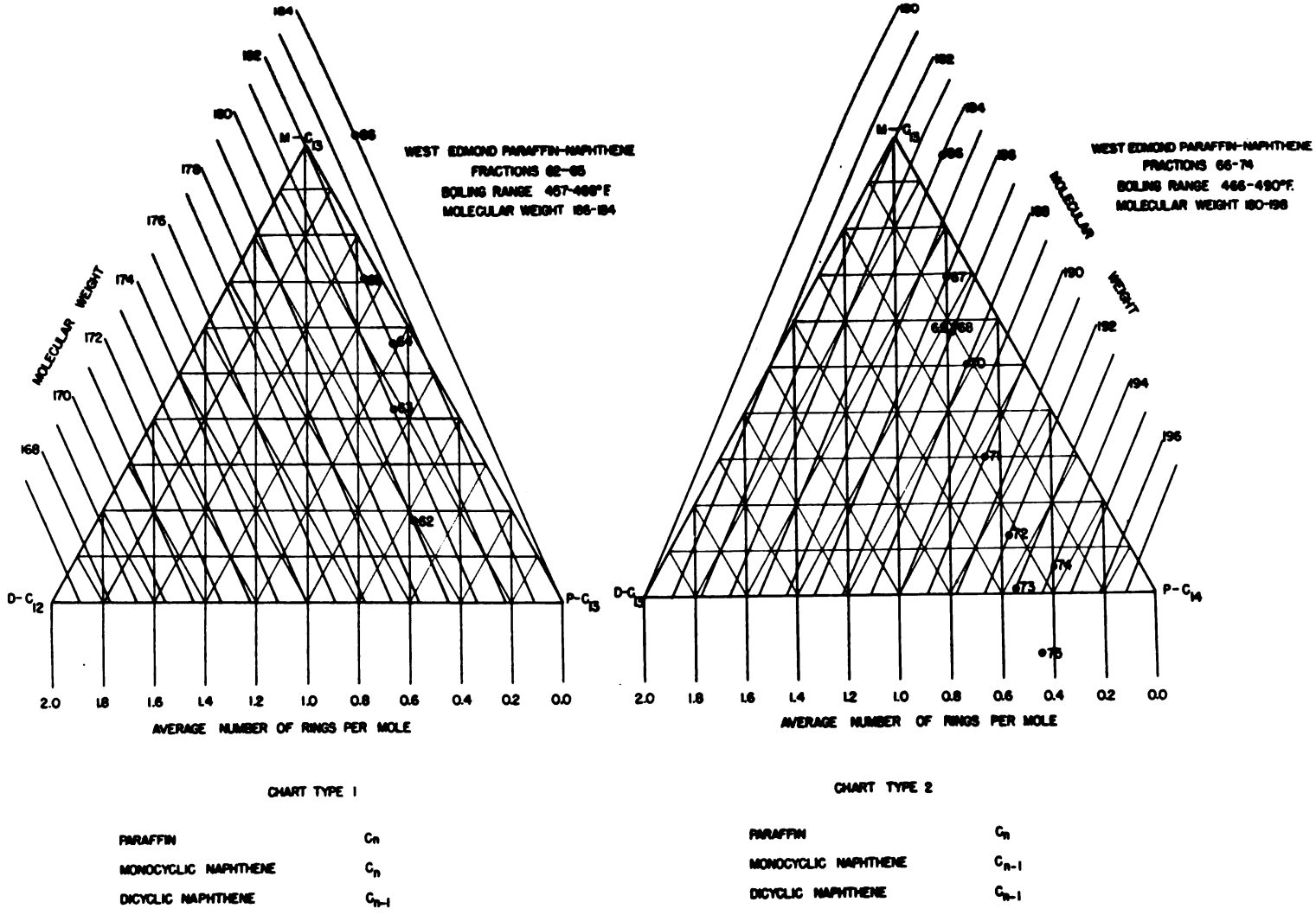

Figure 13. - Ternary composition charts for estimating composition of paraffin-naphthene mixtures.

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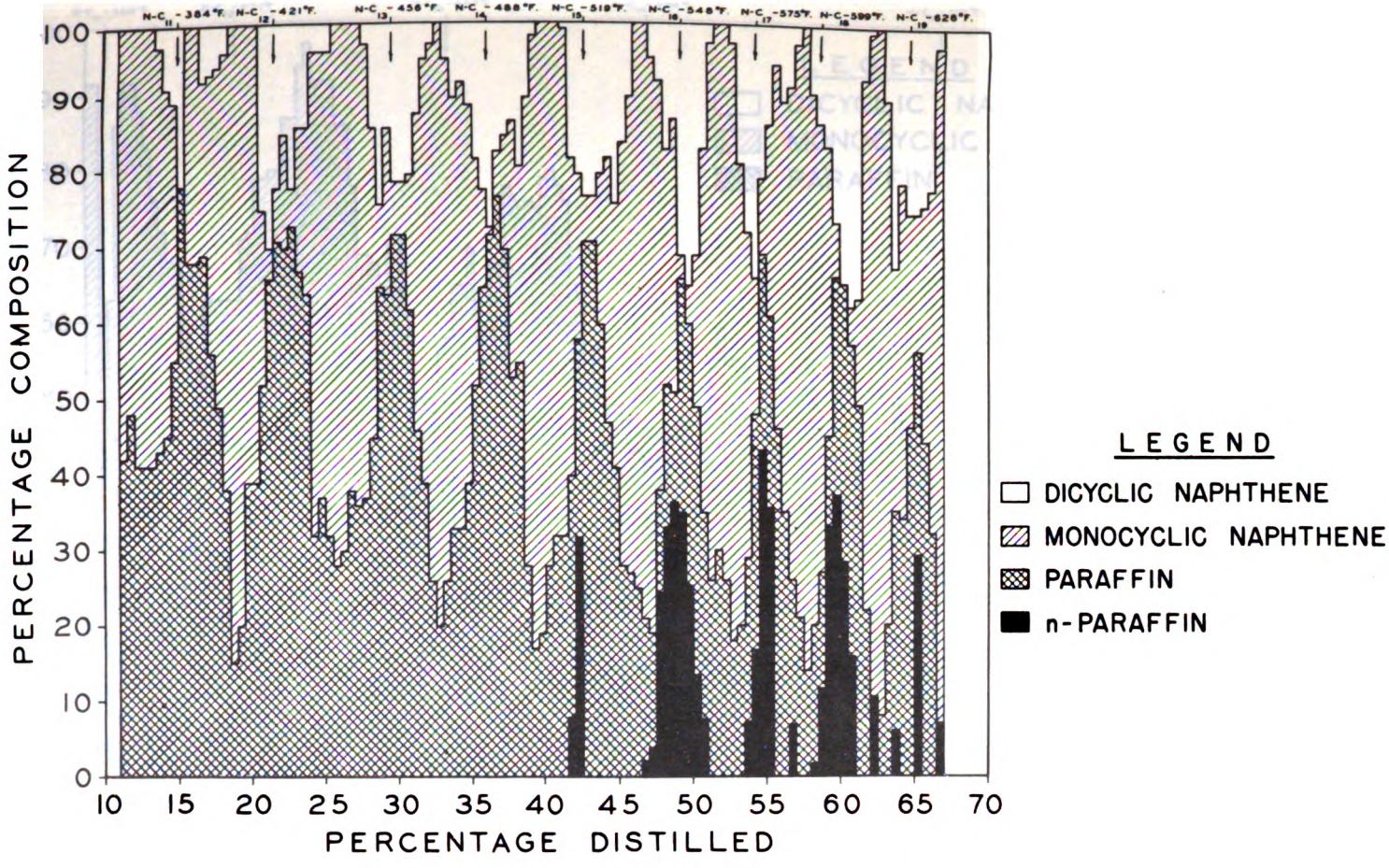

Figure 14. - Composition of paraffin-naphthene portion of West Edmond gas oil.

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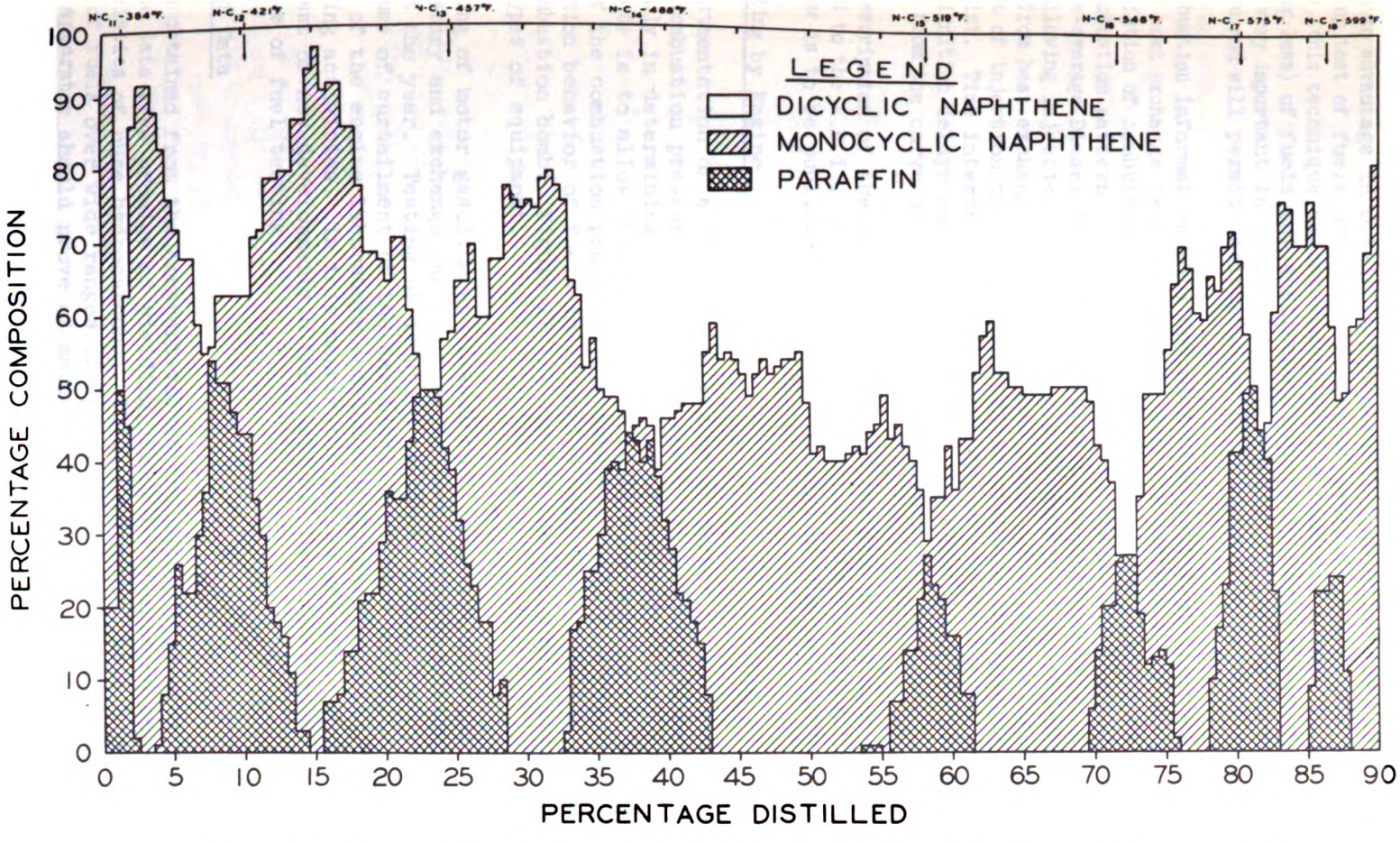

Figure 15. - Composition of paraffin-naphthene portion from catalytically cracked Diesel fuel.

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Combustion Studies with Constant-Volume Bomb

A technique for the investigation of fuel combustion properties by ignition of the fuel in a constant-volume bomb, under development in 1948-49, was further refined and used to advantage in obtaining fundamental combustion information. In addition to permitting test of fuels over wide ranges of pressure, temperature, and oxygen concentrations, this technique allows comprehensive combustion data to be obtained on samples (5 cc. or less) of fuels not available in quantity large enough for engine tests. This is very important in connection with studies of pure hydrocarbons that are very expensive and will permit collection of data impossible to obtain heretofore.

Combustion information obtained with this equipment includes ignition delay and rates of heat exchange between the fuel and the gaseous media both before and after the initiation of combustion. Differences in fuels are exhibited as differences in their combustion patterns. Figure 16, showing characteristic records for poor, medium, and above-average Diesel fuels, illustrates these differences. The pressure decrease shown following injection in these records is caused by a temperature decrease that results from heat exchange to the injected fuel. Ignition delay can be measured from the start of injection to the point of first pressure increase or to any other selected datum point. Time intervals, in milliseconds, are superimposed on the injection curve, so that ignition delays can be determined. The total pressure at any instant can also be taken from the curve by suitable calibrations.

A description of the bomb technique and results of tests on several fuels were presented to the A.P.I., Division of Refining, at its Cleveland mid-year meeting.[45] This paper is to be published in the proceedings of the Institute.

Fuel Testing by Engine

Instrumentation of a Diesel test engine to permit detailed observation of its internal-combustion pressure changes was completed. This equipment has been used successfully in determining ignition quality of commercial-type fuels, but its primary purpose is to allow observations for collecting data to be used in fundamental studies of the combustion process. The data are taken in a form to allow comparison of combustion behavior of fuels in an engine with their behavior in the constant-volume combustion bomb. Figure 17 illustrates the comparable nature of records from the two types of equipment.

Testing of motor gasoline and Diesel fuel samples, including those processed by the laboratory and exchange samples used to test rating precision, was continued throughout the year. Testing of aviation exchange samples was discontinued early in 1950 because of curtailment of the Bartlesville station work with this type of fuel. Personnel of the engine laboratory continued their participation in local and national fuel-testing activities. In this manner the Bureau and other local laboratories have kept abreast of new developments in the field and have contributed to refinement of the science of fuel testing.

Utility of Data

Data obtained from the constant-volume combustion apparatus together with related engine data give promise of yielding valuable information covering the combustion characteristics of pure hydrocarbons. These fundamental data together with data on behavior of fuels over wide ranges of operating conditions that can be obtained with the bomb apparatus should prove of material benefit in realizing more efficient utilization of petroleum.

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[45] Hurn, R. W., and Hughes, K. J., Constant-Volume Combustion of Fuels: Presented before Division of Refining, American Petroleum Institute, Cleveland, May 4, 1950.

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Representatives of both industry and the military continued to display keen interest in these combustion studies. Industry contributed materially to support of the project during 1949-50 by means of a cooperative agreement between the Coordinating Research Council, Inc., and the Bureau.

Fuel Surveys

Reports were published during the fiscal year showing the results of two motor-gasoline surveys[46/47] and one aviation-gasoline survey.[48] These surveys, conducted in cooperation with the American Petroleum Institute and the Coordinating Research Council, Inc., respectively, have been found very useful to petroleum refiners, engine manufacturers, and consumers who require information on the quality of motor and aviation gasoline.

The octane numbers of motor gasolines have reached new highs, establishing records in the history of the gasoline survey and probably in the petroleum-refining industry. The following tabulation shows the national average octane numbers by both the research and motor methods reported during the year and during the previous two years.

Grade of gasoline	Fiscal year 1948		Fiscal year 1949		Fiscal year 1950	
	Summer, 1947	Winter, 1947-48	Summer, 1948	Winter, 1948-49	Summer, 1949	Winter, 1949-50
Research octane number:						
Premium price	85.9	86.2	86.1	86.5	88.0	89.0
Regular price	80.2	80.2	80.1	80.5	81.9	82.4
Motor octane number:						
Premium price	79.2	79.4	79.5	79.7	80.4	81.2
Regular price	75.1	75.3	75.2	75.6	76.4	77.1

The steady increase in antidetonation values is generally credited to two factors: The greater availability of tetraethyl lead (TEL) and the installation of catalytic cracking facilities in more refining plants. Other properties of motor fuels, such as vapor pressure and distillation temperatures, have not varied greatly, although the trend has been toward increased volatility in the first 10 percent evaporated and slightly decreased volatility in the last 10 percent.

Based on inspection data from 16 manufacturers on their products, aviation gasolines produced in October 1949 did not vary significantly in character from those of January 1949 and several preceding years. Some of the results of the aviation-gasoline survey are shown in a summarized form in table 1.

The method used for precision vapor-pressure determination for aviation gasoline, originated at the National Bureau of Standards and further developed by the Bureau of Mines, used in all surveys except the one for 1950, was described in a published report.[49] The 1950 survey was based on data reported to the Bureau of Mines, as are all the motor-gasoline surveys.

46/ Blade, O. C., National Motor-Gasoline Survey, Summer 1949: Bureau of Mines Rept. of Investigations 4644, 1949, 32 pp.

47/ Blade, O. C., National Motor-Gasoline Survey, Winter 1949-50: Bureau of Mines Rept. of Investigations 4702, 1950, 32 pp.

48/ Blade, O. C., National Annual Survey of Commercial Aviation Gasoline, October 1949 Production: Bureau of Mines Rept. of Investigations 4664, 1950, 14 pp.

49/ Holliman, W. C., and Barker, M. G., A Manometric Precision Procedure for Determining the Vapor Pressure of Aviation Gasolines: Bureau of Mines Rept. of Investigations 4635, 1950, 17 pp.

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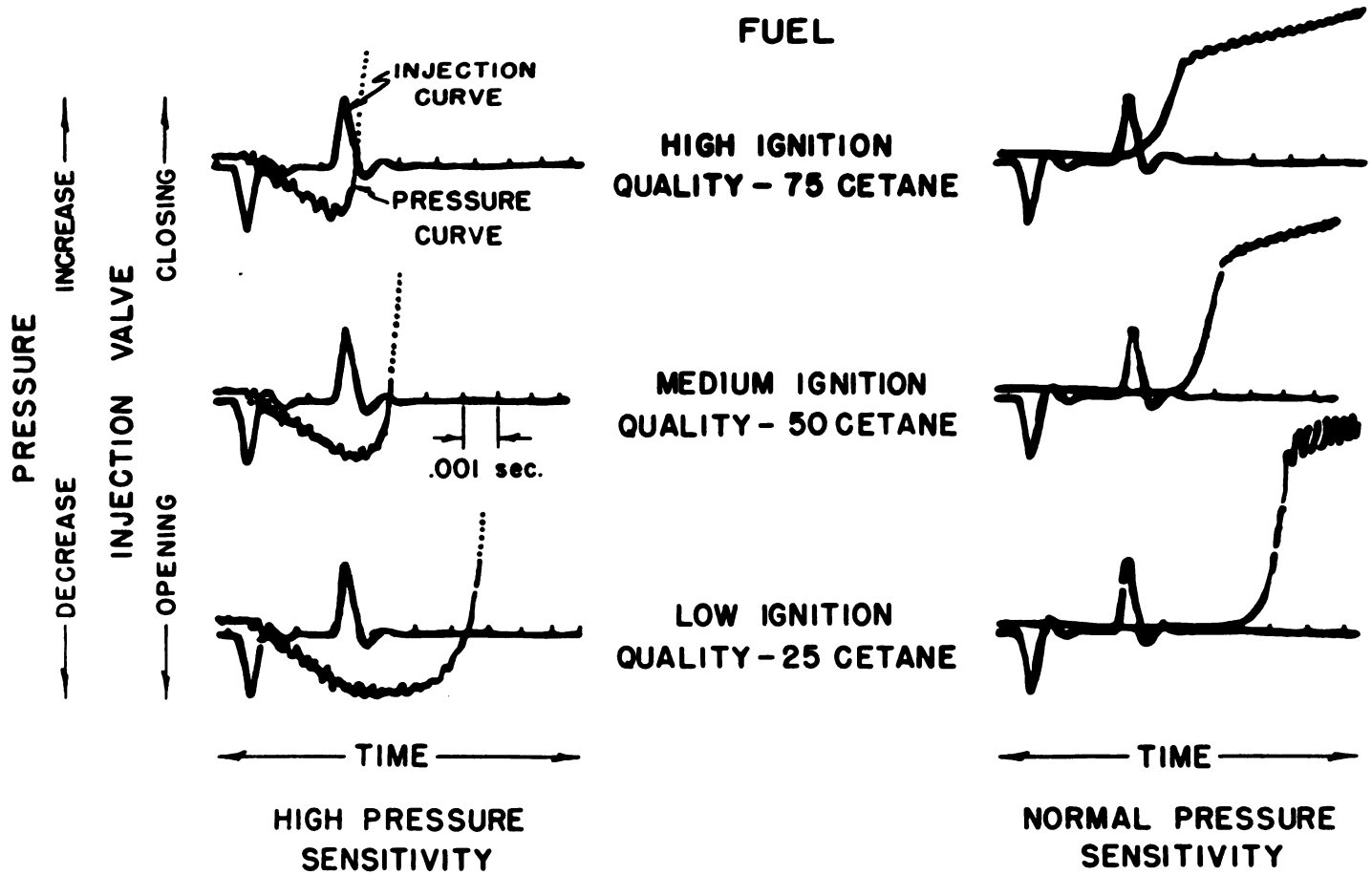

Figure 16. - Constant-volume combustion records.

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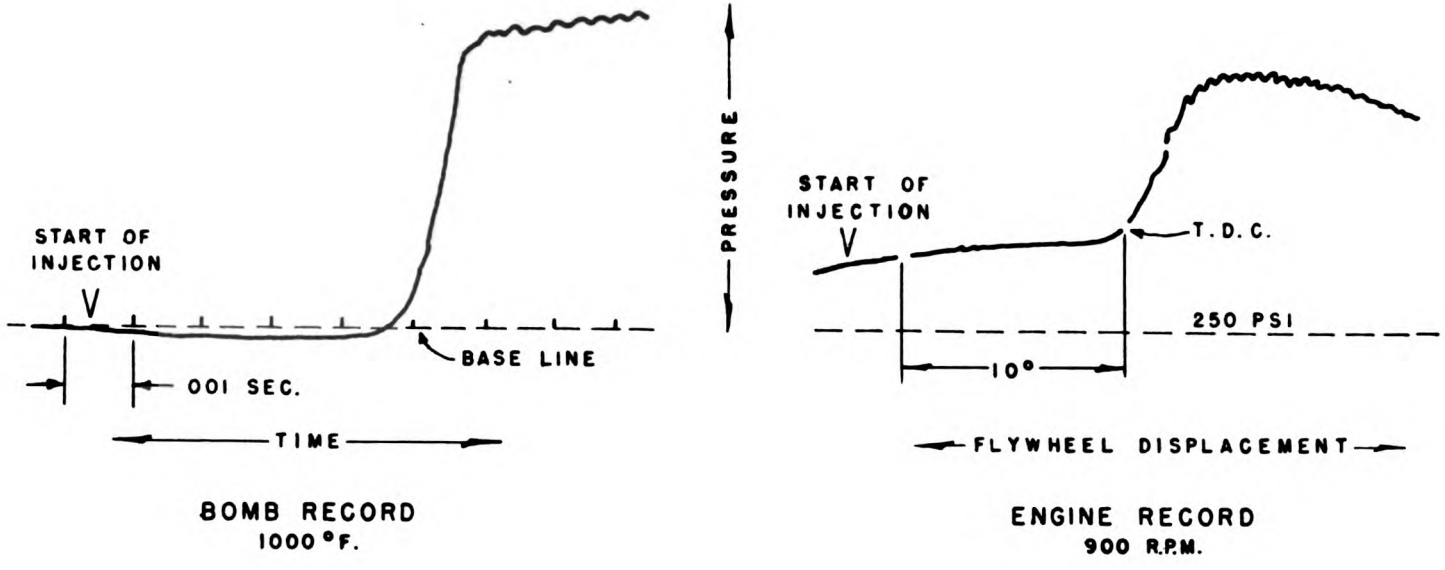

Figure 17. - Characteristic bomb and engine combustion records.

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TABLE 1. - Summary of aviation-gasoline survey data

Test	Grade 80			Grade 91/98			Grade 100/130			Grade 115/145		
	July 1948	Jan. 1949	Oct. 1949	July 1948	Jan. 1949	Oct. 1949	July 1948	Jan. 1949	Oct. 1949	July 1948	Jan. 1949	Oct. 1949
Octane number:												
Lean mixture.....	80.7	80.5	81.1	93.4	93.3	92.8	Iso+ 0.13	Iso+ 0.11	Iso+ 0.14	Iso+ 0.54	Iso+ 0.63	Iso+ 0.57
Rich mixture.....	-	-	87.6	98.8	98.6	98.9	Iso+ 1.33	Iso+ 1.36	Iso+ 1.33	Iso+ 2.95	Iso+ 2.95	Iso+ 2.93
Performance number:												
Lean mixture.....	-	-	-	-	-	-	104.8	104.5	105.3	116.7	118.8	117.4
Rich mixture.....	-	-	-	-	-	-	130.6	131.1	130.6	146.2	146.2	146.1
R.V.P. pounds.....	6.3	6.2	6.2	6.5	6.4	6.4	6.4	6.3	6.7	6.4	6.3	6.5
D86 dist. temp., °F.												
10% evap.	148	149	147	147	149	148	148	148	146	153	151	151
50% evap.	193	194	194	194	193	192	202	202	204	211	209	211
90% evap.	236	239	233	236	237	233	245	246	242	243	242	245
Sp. gr., 60°/60° F.	.706	.707	.705	.710	.712	.713	.706	.708	.707	.701	.697	.700
Visc. kinematic, 70° F., cs.	.594	.595	.616	.591	.592	.616	.604	.601	.628	.619	.616	.640

An annual Diesel-fuel survey was inaugurated in the spring of 1950, and the first published report of this type will appear in the fall of 1950. This is also a cooperative undertaking with the American Petroleum Institute.

Composition and Properties of Asphalts

The remaining experimental data obtained in a survey of straight-run asphalts from Rocky Mountain crude oils were published. The final report[50] on this project compared the yields and properties of asphalts from 20 different crude oils of Wyoming and Montana with asphalts from representative crude oils of California, Arkansas, and Mexico. This comparison indicated that straight-run asphalts from these particular Rocky Mountain crude oils could be used for commercial applications for which asphalts from the representative crude oils had proved to be satisfactory.

Laboratory studies during the past year were related to the preparation of an alumina having constant yet suitable adsorption characteristics for separating the oil and resin fractions of asphalts. Different lots of commercial aluminas were found to have variable adsorption characteristics, depending upon their contents of alpha and gamma forms of alumina. Several laboratory methods of increasing the degree of adsorption of alpha alumina (the least adsorbent form of alumina) and of decreasing the adsorption of gamma alumina were found to be unsatisfactory owing to difficulties in attaining consistent results. Accordingly, two samples of commercial alumina having different adsorption characteristics were blended to yield an alumina having intermediate adsorption characteristics. This alumina was satisfactory for separating the

50/ Stanfield, K. E., and Hubbard, Rethel L., Asphalts from Rocky Mountain Crude Oils, Laboratory Preparation and Comparison: Bureau of Mines Tech. Paper 717, 1949, 77 pp.

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resins from the oils in an asphalt from Tampico, Mex., crude oil, but, unlike an earlier lot of alumina (which is now unavailable), it was not satisfactory for the more resinous asphalts obtained from Kern River, Calif., crude oil. To date, attempts to prepare a standard alumina adsorbent for the analysis of asphalt have been unsuccessful.

THERMODYNAMICS OF PETROLEUM

The science of thermodynamics has played a basic role in some of the pivotal discoveries of the present era. For example, the manufacture of synthetic ammonia, a cornerstone in the agricultural economy, is based on thermodynamic considerations.

Each year chemists and engineers improve the quality and variety of products the petroleum industry supplies to the American public and the Armed Forces. The Bureau of Mines contributed to this steady development of petroleum products by maintaining a well-equipped thermodynamic laboratory at Bartlesville, Okla., devoted to the measurement of the fundamental properties of hydrocarbons and related substances. The data obtained in this laboratory are the basic facts needed by industrial scientists for the invention of new and the improvement of old products. During the past year a program, supported in part by the American Petroleum Institute, to obtain more knowledge of the properties of the many sulfur compounds found in petroleum made good progress. In addition, valuable contributions were made to the knowledge of the thermodynamic properties of pure hydrocarbons. This information was published in technical journals and in the publications of the American Petroleum Institute. The accomplishments of the several different types of thermodynamic investigations are briefly described in the following paragraphs.

Low Temperature Calorimetry

Knowledge of the low-temperature heat capacities, heats of transition, heats of fusion, and melting points, permits evaluation of the entropies of compounds studied, and these, in turn, are valuable factors in calculating the possible economic returns from proposed industrial processes. During the past year a number of compounds were investigated over the temperature range 11 Kelvin (-440° F.) to room temperature.

The results of a study of 2,3-dithiabutane (dimethyl disulfide) were published[51] in an American chemical journal. Also published in this journal was an article[52] on the low-temperature thermal properties of hydrazine, a substance of great current interest because of its potential use as a rocket propellant. Entropy values and low-temperature properties were determined for the sulfur-containing substances 2-thiabutane, 3-thiapentane, ethanethiol, pentanethiol, and 3,4-dithiahexane. The results of a comprehensive study of the thermodynamic properties of nine straight-chain paraffin hydrocarbons have been incorporated in the tabulations issued by A.P.I. Research Project 44.[53]

51/ Scott, D. W., Finke, H. L., Gross, M. E. Guthrie, G. B., and Huffman, H. M., 2,3:Dithiabutane: Low Temperature Heat Capacity, Heat of Fusion, Heat of Vaporization, Vapor Pressure, Entropy, and Thermodynamic Functions: Jour. Am. Chem. Soc., vol. 72, 1950, p. 2424.

52/ Scott, D. W., Oliver, G. D., Gross, M. E., Hubbard, W. N., and Huffman, H. M., Hydrazine: Heat Capacity, Heats of Fusion and Vaporization, Vapor Pressure, Entropy and Thermodynamic Functions: Jour. Am. Chem. Soc., vol. 71, 1949, p. 2293.

53/ Selected Values of Properties of Hydrocarbons: American Petroleum Institute Research Project 44 on the collection, analysis, and calculation of data on the properties of hydrocarbons.

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Vapor Heat Capacity and Heats of Vaporization

The usefulness of data obtained at low temperatures may be extended to the gas phase and to higher temperatures by measuring the heat capacity of hydrocarbon vapors and the heat of vaporization. A study of the above two properties for the five isomeric hexanes was brought to a successful conclusion.[54] The results of measurements on three other important hydrocarbons were incorporated in another publication.[55] Special apparatus had to be designed and built to make measurements on the corrosive sulfur compounds being studied as a part of A.P.I. Research Project 48A.

Combustion Calorimetry

The data obtained in these studies, when coupled with entropies, give positive information about the economic feasibility of projected processes. Because of their industrial importance, efforts are being made to develop suitable techniques for the combustion of sulfur- and chlorine-containing compounds. The problems involved are many and difficult. A rotating bomb designed to overcome some of the major difficulties is now in operation.

Vapor Pressure

A knowledge of vapor pressure as a function of temperature is essential for a complete thermodynamic description of a hydrocarbon or related substance in the region where liquid and vapor may coexist. For this reason, measurements have been carried out on 1-pentene,[55] 2-methyl-1-butene,[55] 2-methyl-2-butene,[55] hydrazine,[52] 2-thiabutane, 3-thiapentane, 2,3-dithiabutane,[51] ethanethiol, 2-methyl-2-propanethiol, and pentanethiol. The difficulties of carrying out the measurements on the three last-named corrosive substances were overcome by the devising of new types of apparatus.

Statistical Mechanical Calculations

In the work on thermodynamics the emphasis is on obtaining fundamental data, by direct measurements, of such accuracy as to be authoritative. However, direct experiments can be applied only over a limited temperature range. Fortunately there are indirect methods of obtaining the thermodynamic properties of many hydrocarbons. Using spectroscopic and other molecular data in conjunction with the theories and computational methods of statistical mechanics, thermodynamic functions have been calculated up to very high temperatures for hydrazine,[52] 2,3-dithiabutane,[51] and 2-methyl-2-butene.[55]

TRANSPORTATION OF NATURAL GAS

The facilities and experience provided by the Bureau's helium-production activities furnish an excellent background for applied engineering research on natural-gas production, transmission, processing, and related instrumentation. Three projects

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[54] Waddington, Guy, Smith, J. C., Scott, D. W., and Huffman, H. M., Experimental Vapor Heat Capacities and Heats of Vaporization of 2-Methylpentane, 3-Methylpentane and 2,3-Dimethylpentane: Jour. Am. Chem. Soc., vol. 71, 1949, pp. 3902-3906.

[55] Scott, D. W., Waddington, Guy, Smith, J. C., and Huffman, H. M., Thermodynamic Properties of Three Isomeric Pentenes: Jour. Am. Chem. Soc., vol. 71, 1949, p. 2767.

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have been worked on during the fiscal year 1950 at the Bureau of Mines Amarillo, Tex., helium plant. All have been cooperative projects with the Natural Gas Department, American Gas Association. These are: (1) the removal of nitrogen from natural gas, (2) the development of a dew-point recorder for natural gas, and (3) the study of pipeline flow.

Removal of Excess Nitrogen from Natural Gas

Nitrogen in natural gas is worthless as a fuel component; further, it costs as much to transport this constituent as it does to transport the combustible components of natural gas. If the nitrogen content of the gas is unusually high, the resulting low B.t.u. content or heating value may prevent the ready sale of the gas. Previous work on this problem has indicated that liquefaction and fractionation might be the most favorable method of nitrogen removal. Research was continued on a nitrogen-removal pilot unit operated at the Bureau's Exell helium plant near Amarillo, Tex., where facilities, experienced personnel, and other conditions were highly favorable for experimentation of this type.

The processing cycle was revised somewhat, and a series of runs was made to obtain data with respect to fractionation, enthalpy, and other factors. Many gas samples were taken during these operating periods, and they were analyzed later with the mass spectrometer. These analyses, together with the temperatures and pressures that prevailed within the equipment at the time the respective samples were taken, are being used to determine phase equilibria, which are basic data needed for design of equipment and efficient operation of the process for removing nitrogen from natural gas by liquefaction and fractionation. Data for phase-equilibria determinations were taken over the ranges of 0° F. to -240° F. in temperature, and 100 to 600 lb. per sq. in. absolute in pressure.

The interest of the natural-gas-transmission industry has decreased in regard to the nitrogen-removal problem. Therefore, the committee of the American Gas Association having cognizance of this problem recommended that active research be discontinued for the time being and that the results of the study be summarized. The committee expressed the opinio that the research can be started again whenever enough interest is shown to justify reacti vation. One or more reports will be prepared to discuss gas-separation processes for removing nitrogen from natural gas and to present the technical data obtained from the nitrogen-removal pilot-plant experiments.

Dew-Point Recorder for Natural Gas

Billions of cubic feet of natural gas is transported through long-distance pipe lines daily. Virtually all of this gas is dehydrated before it enters the transmission line. As a means of knowing how much water vapor remains in the gas, the Bureau, with the cooperation of the Natural Gas Department, American Gas Association, has developed an instrument that gives a continuous record of the dew point or water-vapor content of the gas. Tests on natural-gas pipe lines over a period of 18 months have shown this recorder to be dependable and practical.

These tests were on pipe lines transporting natural gas that was free of diethylene glycol and ethanolamine vapors. Glycol is used in some dehydration plants to remove water vapor from natural gas. Amine or mixtures of amine and glycol are used to remove acid gases such as hydrogen sulfide. It was found that amine and glycol vapors in the natural gas presented difficulties in operating the dew-point recorder. However, considerable success was achieved this year in devising a means to trap the glycol vapors and thus prevent them from entering the hygrometer cell of the instrument without changing the water condition of the natural gas. In fact, a recorder is operating on a pipe line carrying

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natural gas treated in a glycol-type dehydration plant, and the calibration of the instrument, expressed in dew-point temperatures, remains satisfactorily constant with only weekly inspections. The amine problem is more difficult, but there were some favorable developments during the year that indicate a practical solution will be found.

The dew-point recorder has been completely unitized and mounted in a cabinet (fig. 13). Much interest has been manifested by the natural gas industry in this instrument, which indicates that it will serve a highly useful purpose in checking the performance of dehydration plants to prevent faulty operation and thus safeguard the operation of the transmission systems.

A paper[56/] on apparatus and procedures for obtaining dew-point data, means for obtaining the water-vapor content from those data, and instruments for giving a continuous record of the dew point of gas was given to an industry group attending a course in gas technology.

Flow of Natural Gas in Pipe Lines

With the higher pressures and larger-diameter pipe lines now being used for gas transportation, further information on pipe-line flow is needed. A third cooperative project with the Natural Gas Department, American Gas Association, is being carried on by personnel of the Amarillo station of the Bureau of Mines. The objective of this study is the development of an accurate formula for predicting the flow capacity of new lines or measuring the efficiency of old ones. Extensive tests are being made to determine the effect of internal surface roughness on the flowing capacity of pipes.

Some of these tests were made during the winter period of operation of the Goleta, Calif., storage reservoir of the Pacific Lighting Corp. When spring came, causing a reduction in the rate of gas flow, the equipment was left in readiness for reactivation of the work in late fall, when higher flow rates will prevail again. In the intervening period, attention is being given to improvements in instrumentation and modifications of the test runs to expedite the flow experiments when work is resumed at Goleta.

HELIUM-TRACER STUDIES

The Bureau's helium-production program and the research conducted at the Amarillo, Tex., helium plant to ascertain the helium resources of the Nation foster new and expand old uses for helium, and improved plant processes and equipment are reviewed in the Bureau of Mines Minerals Yearbook and other writings. However, the use of helium as a tracer in determining underground movements of oil and gas will be discussed in the following paragraphs because of its special interest to the petroleum industry.

Helium, when used as a tracer gas, is mixed with natural gas being injected into a reservoir for secondary recovery of oil, cycling, pressure maintenance, or other purposes. To supplement information from earlier tests, a cooperative helium tracer-gas project was conducted in the Pure Oil Co.'s Cabin Creek, W. Va., field. A report[57/] was completed giving the results obtained in three separate experimental helium tracer-gas tests that were run during this study.

56/ Deaton, W. M., Determination and Recordings of Dew Points in Natural Gas Systems: Proceedings of the 5th Annual Short Course in Gas Technology, Texas College of Arts and Industries, Kingville, Tex., November 1950.

57/ Frost, E. M., Jr., Helium Tracer-Gas Studies in the Cabin Creek, W. Va., Oil and Gas Field: Bureau of Mines Rept. of Investigations 4715, 1950, 28 pp. Also, Producers Monthly, vol. 14, No. 11, Sept. 1950, pp. 36-39; vol. 14, No. 12, Oct. 1950, pp. 31-35; vol. 15, No. 1, Nov. 1950, pp. 39-42; vol. 15, No. 2, Dec. 1950, pp. 29-34.

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In one of these tests, tracer helium was added to the injected gas in a concentration of 1 percent for a 1-week period. In the second test, tracer helium was added at another and relatively remote well in a concentration of 5 percent for approximately 4 days. In the third test, a slug of essentially pure helium was injected as a tracer at the well used in the first test, but after the effects of the first test had almost subsided. The quantities of helium injected in the first and third tests were approximately the same.

As in earlier experimental work in other fields, these tests gave data concerning the direction and rate of travel of the injected gas, the proportion of injected gas in the gas produced from the various wells, and the nature of the transition from formation gas to injected gas in the production from the wells. In the vicinity of one of the test wells, the rate of travel of injected gas in one direction was approximately twice that in the opposite direction. The tests showed that a wave of tracer-laden gas formed by injecting helium for a relatively short period will give substantially the same information that might be obtained by injecting helium continuously until the helium content of the produced gas reaches a stable maximum value.

The report on the Cabin Creek helium-tracer studies was presented at a meeting of the Eastern District of the American Petroleum Institute's Topical Committee on Production Technology on June 23, 1950.

The Bureau conducted ihe first few helium tracer-gas projects. The report on the tests in the Cabin Creek field gives the findings of one of those projects. Now the Bureau provides only an advisory engineering service, which is available to operators who conduct their own helium tracer-gas projects. During the year, one project was started in California and arrangements were made for another in West Texas.

EFFECTS OF PROGRAM

The Bureau's research on petroleum and natural gas is directed primarily toward conservation through increased efficiency. In its studies of primary production, the Bureau seeks ways of using the natural energy inherent to the producing formations to recover greater quantities of oil. In its work on secondary recovery it endeavors to find more efficient and effective methods of introducing energy from the surface to supplement the natural energy in driving oil to producing wells. In summary, these two phases of the program are aimed toward developing methods of producing oil and gas at rates required to meet the Nation's needs and ultimately to recover the maximum practicable quantity, or, conversely, to leave a minimum beyond recovery when the economic limit forces abandonment of a field.

The major objective of the studies of petroleum chemistry and refining and of petroleum thermodynamics is to supply basic information that will foster the manufacture of better products in larger quantities from the oil and gas produced and lead to greater efficiency in their utilization. The research on transportation of natural gas points to ways of decreasing the energy (fuel) required to move the gas through the lines, conserving steel in their construction, and improving service.

Thus, through this research program, conducted in close cooperation with industry, the Bureau is increasing the quantity and quality of petroleum products that will be available to the Nation's citizens in future years.

Int. - Bu. of Mines, Pgh., Pa.

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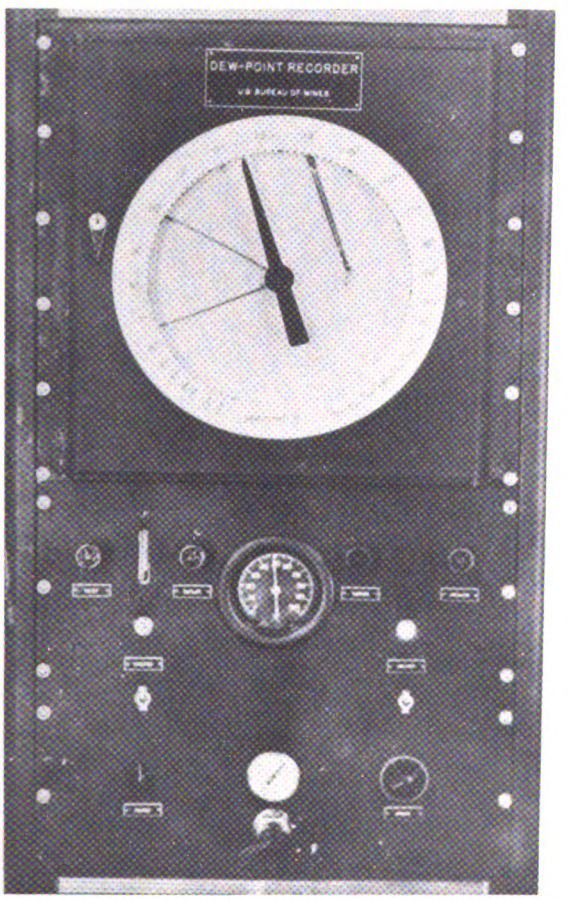

Figure 18. - Bureau of Mines dew-point recorder.

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